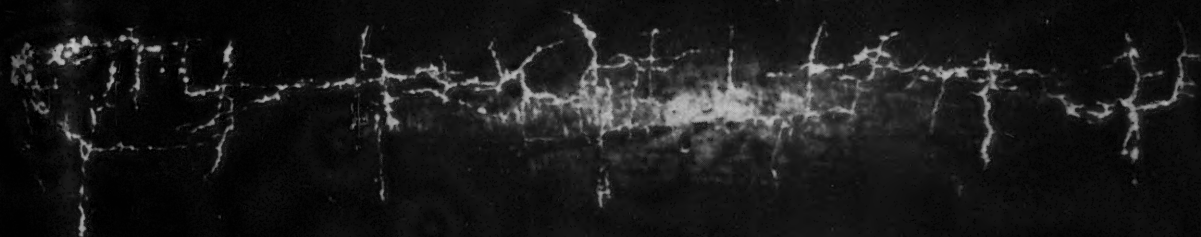


# *Corrosion*



# CORROSION

## affects every industry

### BILLIONS OF DOLLARS ARE LOST ANNUALLY

And your plant is not immune. Corrosion may attack in many ways...

#### 1 Your Hard-to-Get Equipment

... may be ruined by constant attack of acids, alkalies, fumes, chemicals, moisture, gases and weathering.

#### 2 Your Valuable Products

... are subject to contamination and loss whenever they are in contact with corroded surfaces.

#### 3 Expensive Shutdowns

... and loss of production with costly man-hours for repairs may be incurred unnecessarily.

#### 4 Your Profits

Ruined equipment, contaminated products, the loss of production and repair expense caused by shutdowns mean only one thing... reduction in *your* profits!

RIGHT NOW... you should investigate the economies of using Amercoat; a *line* of coatings built to protect against *specific* industrial corrosion hazards.

Our field engineers are available... without obligation... to check your plant for corrosion problems... and to give you *specific* recommendations.

FOLLOW THE LINE OF *max* RESISTANCE... PROTECT WITH *Amercoat*!

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A division of American Pipe and Construction Co.

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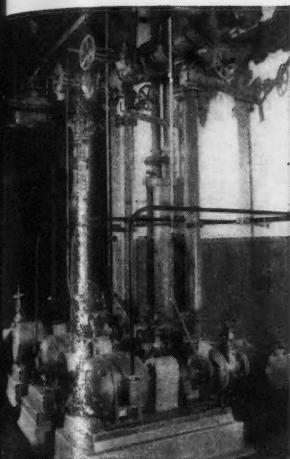
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The tougher the valve  
the better in making  
rayon textiles



To produce the quality rayon made by Industrial Rayon Corporation's Painesville, Ohio, plant requires the handling of some mighty corrosive solutions in vital continuous processing operations.

That's where four hundred corrosion-resistant ALOYCO gate valves, like you see above, play a vital role... operating without maintenance, though many were installed three and a half years ago.

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May we suggest that you call our Aloyco Corrosion Clinic, at no obligation, for expert consultation and engineering service. It is available to you for "diagnosis" and "treatment" of problems involving valves vs. corrosives.

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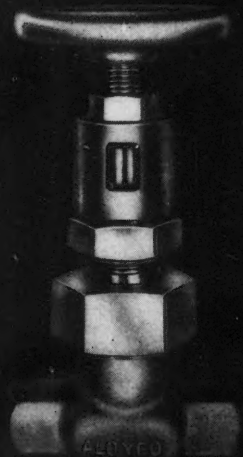
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**ALOYCO VALVES**  
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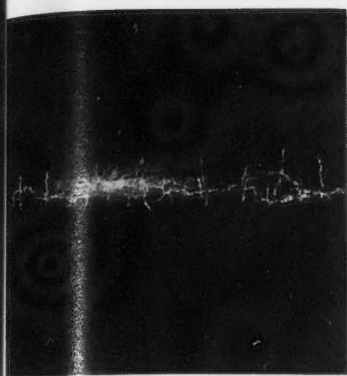
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THIS MONTH'S COVER—Zygo, a fluorescent penetrant developed by Magnaflux Corp., Chicago, has indicated here corrosion cracks in a nickel oil refinery still tube. It was known several tubes in the still were leaking and the fluorescent penetrant permitted identification of the faulty ones. This is an illustration from a technical paper presented at South Central Region's New Orleans meeting on the uses of this inspection method.



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research and control

Published monthly as its official journal, by the National Association of Corrosion Engineers, Inc., at Houston, Texas, U. S. A., as a permanent record of progress in corrosion control.

Vol. 8

OCTOBER, 1952

No. 10



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- Authors of technical papers on corrosion are invited to submit titles and abstracts of papers to appropriate officials of the Technical Program for the 1953 Chicago Conference of the National Association of Corrosion Engineers, to be held March 16-20.
- Information on symposia titles and the names of symposia chairmen named to date will be found in the News Section of the October issue of Corrosion.
- The Association also welcomes at any time constructive discussions of its technical material and technical papers on corrosion for publication in Corrosion.
- Interested authors may secure on request a copy of the "NACE Guide for the Preparation and Presentation of Papers" which describes the Association's customs and needs more fully.

## ***Inco High Temperature Research Note:***

# **CARBURIZATION**

### **...and its effect on metals at high temperatures**

In chemical processing, petroleum refining, steam power generation and other work at high temperatures, metals are often exposed to atmospheres that may have unsuspected corrosive effects.

Common soot, itself, can seriously damage the usefulness of some heat-resisting alloys. When carbon diffuses into the grain boundaries, it embrittles the metal.



Under the microscope, this specimen reveals the reason why a carburizing box broke after 2200 hours of service at 1860° F. The thick black lines are the hard carbides which intruded along the grain boundaries and oxidized, causing embrittlement of the metal.

From INCO Research Laboratory, Case Report No. 5082.1.

This is one form of destruction that can be caused by carburization.

It may be a tolerable condition in some applications. Where the metal is subjected to impacts or sudden temperature changes, however, carburization can be the reason for premature failure.

The susceptibility of an alloy to damage by carburization seems to depend on the relative amounts of iron, chromium and nickel in the alloy. Nickel resists carburization. As a result, nickel-base alloys show excellent resistance to this form of high temperature corrosion.

There are some additions to this general observation:

For example, high chromium steels with a ferritic structure do give good resistance to carbide formation. The practical limit on their usefulness, however, is the loss of strength they suffer at temperatures above 1300° F.

With exceptions like this in mind it is apparent why a considerable background of information is necessary to predict the performance of alloys at high temperatures.

That is why INCO'S metallurgists are continually investigating the behavior of alloys in the field and in research laboratories at Bayonne, N. J., and Huntington, West Va., to make more of this information available to industry through Inco's

High Temperature Engineering Service.

Their creep and rupture tests at temperatures as high as 2100° F. supply data on the strength properties of high nickel alloys. Their studies of corrosive attack by various hot atmospheres, fused salts and molten metals provide the type of information that helps industry to select more suitable, longer-lasting materials for various high temperature applications.

When high temperature performance is a problem to you (or when you expect it to be), Inco High Temperature Engineers may have the answer for you. They have devised a special High Temperature Work Sheet on which you can set down your problem. Write for your copy now.



Send for your copy of this new High Temperature Work Sheet; it simplifies gathering the full story of your problem.



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67 Wall Street, New York 5, N. Y.



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Send for this new booklet describing Alcoa's new aluminum coiled tube—UTILITUBE. Alcoa UTILITUBE is designed for instrumentation, fuel, air and lubrication applications. It is easier to form, flare and bend than annealed copper. It will save you up to 40% on tube costs—gives you 3 times more feet per pound. It comes in standard 50- and 100-foot lengths and in economical, long lengths up to 1,000 feet in some sizes. Write:

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(Continued from Page viii)

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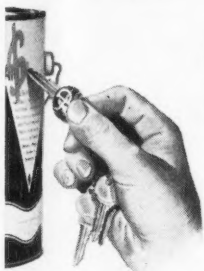
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... to

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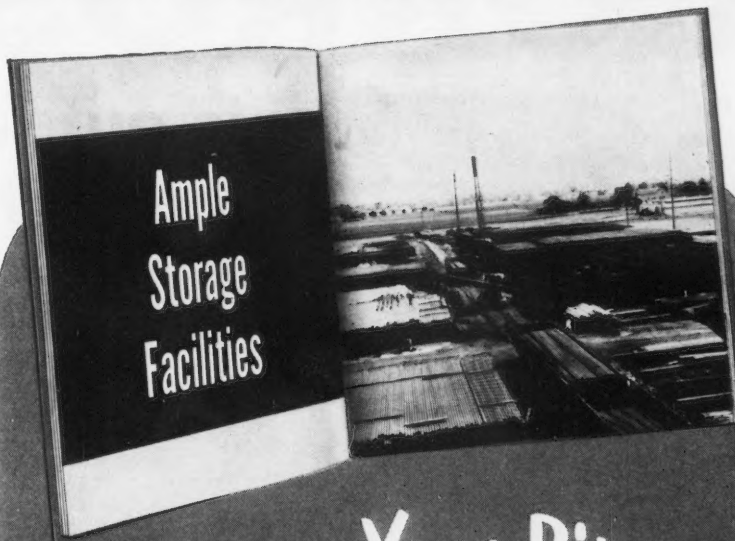
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Until You  
Actually Need It!

Your pipe can be stored by PLS for an unlimited time,\* and at your request, PLS will coordinate the cleaning, priming, coating and wrapping, and reshipment, so that when your job begins, this pipe will be on the job-site—ready to go. \*(Pipe shipped on stop-over in-transit privileges can be stored for the period of one year before reshipment is required.)

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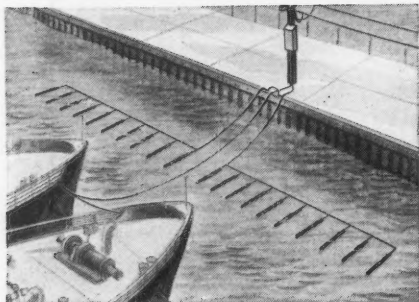
General Office and Plant:  
Franklin Park, Illinois



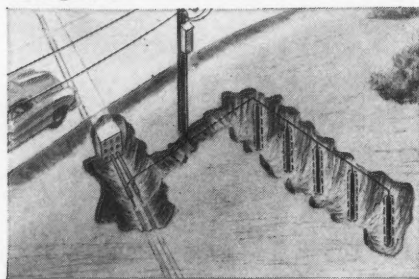
Plants at Glenwillard, Penna.; Longview, Texas;  
Corpus Christi, Texas; Harvey, Louisiana;  
and Sparrows Point, Maryland



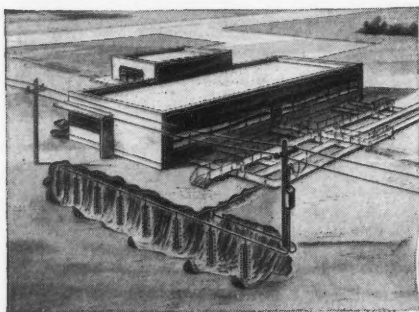
# CORROSION



**SHIP HULLS**—Inactive ship hulls are effectively and economically protected by remotely located "National" Ground Anodes. Active vessels also can be protected with anode plates mounted on or near the bilge keel.



**LEAD-COVERED CABLES**—Both telephone and power cables are cathodically protected in many areas with "National" Anodes.



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...mark phenomenal acceptance of "EVEREADY" No. 1050 Industrial Flashlight Battery by a broad cross-section of industry. Delivering *twice the usable light* of any battery we've ever made before, it will not swell, stick, or jam in the flashlight... has no metal can to leak or corrode.

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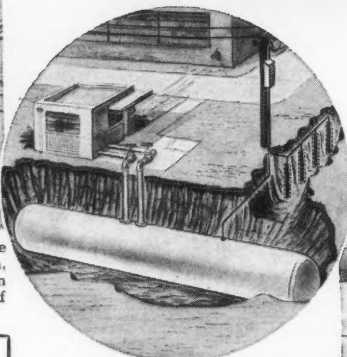
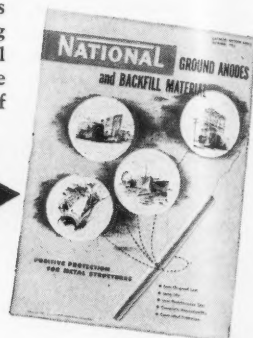
**STOP** it with **NATIONAL**  
TRADE-MARK  
**Graphite Ground Anodes**

You can do much more than *arrest* corrosion. You can *eliminate it completely*—at a fraction of the cost of maintenance and replacement—with cathodic protection, employing "National" Graphite Ground Anodes.

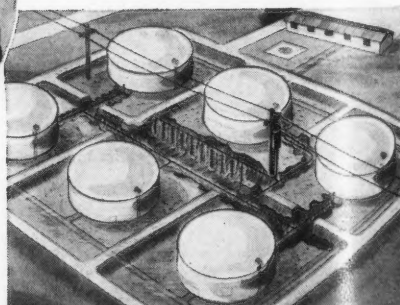
Proved and widely accepted for the protection of steel and cast-iron pipe lines, cathodic protection is finding new applications every day in the scores of industries that otherwise must continue to pay their share of the terrific toll levied against metal structures by soil, air, water and dissolved minerals.

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WRITE FOR  
CATALOG SECTION S-6510



**INDUSTRIAL PLANTS**—External corrosion of buried or submerged metal structures can be completely eliminated by the use of "National" Ground Anodes.

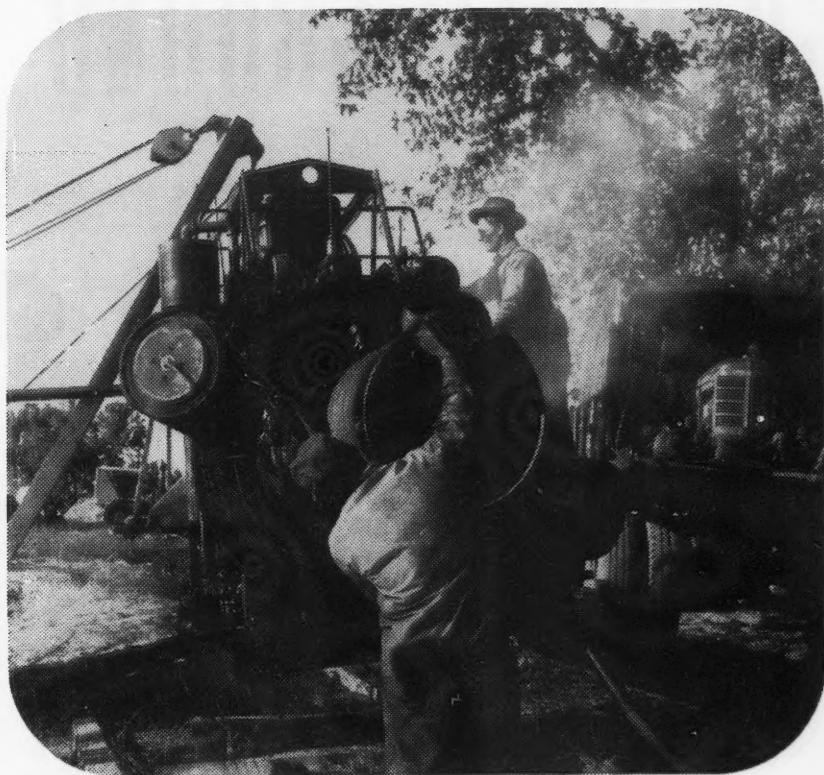


**TANK FARMS**—The cost of a complete "National" Anode installation for the protection of a tank farm, including the cost of rectifiers, is frequently paid for within a very few months with savings from reduced structure maintenance.

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Pipeline crews really *roll* when they start coating with Pitt Chem Modified Enamel. That's because every ton of this impervious protective coating provides the same peak application characteristics and excellent bond as the last. Pitt Chem Modified Grade heats up faster, flows better from kettle to pipe and coats more pipe per ton because of its consistent top quality.

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CORROSION—October, 1952

CATHODIC PROTECTION SERVICE PROUDLY



# *Announces* The New CPS GRAPHITE GROUND ANODES

*\*with Super-Strong Leads!*  
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FROM OUR LARGE STOCKS

\*Normally supplied with 3 ft. No. 6 direct burial cable but we will attach leads to your specifications.

Cathodic Protection Service, through a nationwide agreement with GREAT LAKES CARBON CORPORATION, now offers the NEW C P S—Graphite Ground Anodes as the latest addition to its growing family of cathodic protection materials and supplies.

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Everything in the Cathodic protection field . . .  
from an insulating washer to a turnkey contract installation



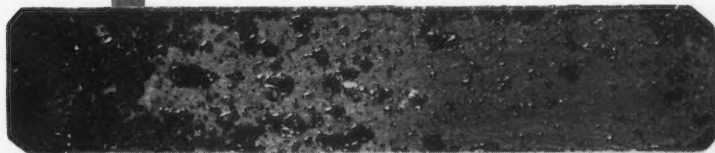
**SANTOLENE C PROVED** its effectiveness in this test. Metal strips illustrated were immersed in gasoline samples containing various amounts of Santolene C. 1—Control, containing no Santolene C. 2—Strip from sample containing Santolene C at the rate of 3 pounds per 1,000 barrels. 3—Strip from sample containing Santolene C at the rate of 5 pounds per 1,000 barrels. 4—Strip from sample containing Santolene C at the rate of 7½ pounds per 1,000 barrels. If you want to make a similar test with your own products, we will be glad to supply a sample of Santolene C.

Results of

## SANTOLENE C

are easy

to see



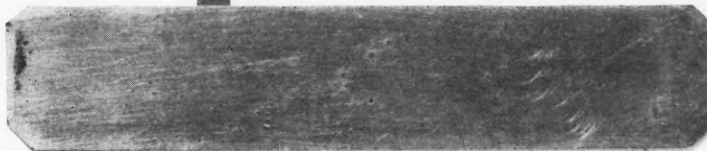
1



2



3



4

Monsanto Santolene C, added to light petroleum products, controls corrosion. Take a look at the metal strips illustrated here and you'll see the results of Santolene C applied at various concentrations.

In addition to convincing proof in extensive tests, Santolene C has demonstrated its effectiveness in

service. It has been used by major oil companies for more than two years in all types of light petroleum products.

Santolene C is product-soluble. It burns completely with fuel, leaving no residue. Tests in both gasoline and diesel engines show that the additive does not reduce engine cleanliness, causes no injector fouling, has no adverse effect on engine performance.

If corrosion is a problem in your storage or transportation of light petroleum products, investigate Santolene C. It may be your solution. Write for a copy of Monsanto Technical Bulletin No. O-62, "Santolene C," which contains information that may be worth money to you. MONSANTO CHEMICAL COMPANY, Organic Chemicals Division, 1700 South Second Street, St. Louis 4, Missouri.

*Santolene: Reg. U. S. Pat. Off.*

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CHEMICALS AND PLASTICS

SERVING INDUSTRY... WHICH SERVES MANKIND

# THE NATIONAL ASSOCIATION OF CORROSION ENGINEERS

is a non-profit, scientific and research association of individuals and companies concerned with corrosion or interested in it, whose objects are:

- (a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.
- (b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.
- (c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.
- (d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.
- (e) To promote standardization of terminology, techniques, equipment and design in corrosion control.
- (f) To contribute to industrial and public safety by promoting the prevention of corrosion as a cause of accidents.
- (g) To foster cooperation between individual operators of metallic plant and structures in the joint solution of common corrosion problems.
- (h) To invite a wide diversity of membership, thereby insuring reciprocal benefits between industries and governmental groups as well as between individuals and corporations.

It is an incorporated association without capital stock, chartered under the laws of Texas. Its affairs are governed by a Board of Directors, elected by the general membership. Officers and elected directors are nominated by a nominating committee in accordance with the articles of organization. Election is by the membership.

Inquiries regarding membership, and all general correspondence should be directed to the Executive Secretary at the administrative headquarters of the National Association of Corrosion Engineers at 1061 M & M Building, No. 1 Main Street, Houston 2, Texas.



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## Symposium on SULFIDE STRESS CORROSION

The papers presented herein include an introductory paper reprinted from the Canadian Mining and Metallurgical Bulletin and four papers presented at the Oil and Gas Industry Symposium on Sulfide Stress Corrosion on March 12, 1952 during the Eighth Annual Conference and Exhibition of the National Association of Engineers at Galveston, Texas.

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# Hydrogen Sulphide Corrosion Cracking of Steel\*

By L. W. VOLLMER\*

## Introduction

**D**URING the past three years at least fifty strings of 9 percent nickel steel tubing have exhibited excellent resistance to the variably severe corrosive conditions typical of many of the sweet condensate wells in Mississippi, Louisiana, and Texas. This experience, and the characteristically outstanding performance of other nickel alloy steels under hydrogen sulphide corrosive conditions, dictated the selection of 9 percent nickel steel tubing for the second well drilled in the sour condensate Pincher Creek field of southwestern Alberta. The damaging effects of the Pincher Creek reservoir fluids on well equipment had been manifested in the discovery well by the rapid and not yet fully understood failure of API Grade N-80 tubing, stainless steel wire lines, and alloy steel fishing tools. Two very unexpected failures in the 9 percent nickel steel tubing, after only six days of service in production tests, instigated an extensive and continuing investigation to ascertain the probable cause of failure. This discussion presents some of the results obtained thus far which cast a little light on the perplexing behavior of steels in hydrogen sulphide environments in general, and in sour condensate wells in particular.

## History of Tubing Failures

Walter Marr well No. 1, in which the 9 percent nickel steel tubing failures occurred, produces from the Madison lime at the depth interval approximately 12,200 to 12,750 feet. The gas-oil ratio is about 26,000 and the gas contains 10.2 percent hydrogen sulphide and 6.3 percent carbon dioxide by volume. During six days of initial testing, the well was produced at an average rate of 7.5 million cubic feet per day, yielding about 300 barrels per day of distillate along with approximately the same volume of formation water. This water, containing some 37,500 ppm dissolved solids, was high in bicarbonates and, of course, hydrogen sulphide. The bottom-hole pressure and temperature were 4,868 psi and 188 degrees F, respectively, at 12,200 feet. The closed-in well-head pressure was 3,236 psig. The well-head pressure at 7.5 million cubic feet per day flow rate averaged 1,350 psig.

The 12,200-foot string of 9 percent nickel steel tubing (2 $\frac{7}{8}$ -in. od, 6.50 lb.) was set on a production packer at 12,186 feet. A 3300-foot string of 5-inch casing was hung in the annulus of the tubing and the 7-inch oil string for circulation of hot Diesel oil (140 to 150 degrees F) down the 2 $\frac{7}{8}$ -inch to 5-inch annulus and up the 5-inch to 7-inch annulus. This raised the well-head flowing temperature to 96 degrees F and successfully prevented hydrate formation.

## Abstract

Sudden blow-out of tubing after only six days of production tests of the 12,000-foot Walter Marr Well No. 1, Pincher Creek Field, Canada caused an investigation of causes for failure. Examination of the removed 9 percent nickel steel tubing showed no obvious defects and calculations showed failures not due to excessive triaxial loading.

Investigators, after discarding embrittlement as a cause, initiated tests to determine if stress corrosion cracking was the cause, because well fluids and gases included 10 percent hydrogen sulfide and 6.3 percent carbon dioxide by volume. Specimens of 9 percent nickel steel tubing were submerged in tap water containing hydrogen sulfide and carbon dioxide in a cell at 5000 psi and 190 degrees F. All specimens failed in six days.

Numerous other tests of the tubing and other steels at varying loadings were conducted, including pre-stressed and re-heat treated but not pre-stressed samples. These tests developed the following tentative conclusions: 1) Tubing failures at the Marr well resulted from stress-corrosion cracking. 2) Susceptibility to failure in hydrogen sulfide environments is not limited to 9 percent nickel steel. Steels treated to produce Rockwell C24 to 26 may be rendered susceptible. 3) Plastic deformation greatly increases susceptibility to failure, but is not essential if protective corrosion products films are removed. 4) Embrittlement is not a primary cause of failure but may be contributory. 5) Susceptibility may be reduced by composition changes.

The first tubing failure occurred while the well was being killed for a cement plug-back to shut off water. Approximately 137 barrels of water at 50 degrees F had been pumped from a nearby lake into the annulus in the process of displacing Diesel oil (343 bbl.) when the well blew out through the water lines. It was found that the top tubing joint had parted 26 feet below the well-head and about 8 inches above the upset taper. Figures 1 and 2 show the failure.

While fishing, the tubing parted again at 1201 feet under a load of 80,000 to 85,000 pounds at approximately the midpoint of the joint. Both halves of this failure are shown in Figure 3.

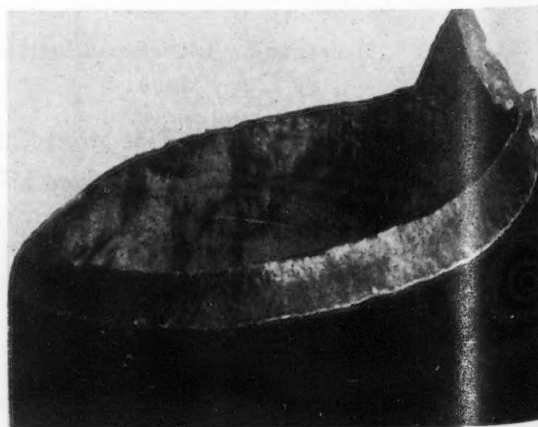


Figure 1—One half of first tubing failure, 26 feet from surface.

\* A paper presented at the Annual Western Meeting, Canadian Institute of Mining and Metallurgy, Calgary, Alta., October, 1951. Reprinted from *The Canadian Mining and Metallurgical Bulletin*, February, 1952. [Transactions, Vol. LV, 89-95 (1952)].

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It is considered significant that, although large segments of both failures are brittle in appearance, there is positive plastic deformation of other segments so that the entire fractures cannot be classed as truly brittle.

After a successful plug-back to about 12,400 feet, the well was acidized and further tested through X-80 tubing with an internal plastic coating. The well was shut in for some 15 months, but recently limited production was resumed to test materials and methods of minimizing corrosion and preventing hydrate formation.

### Analysis of Failures

The recovered 9 percent nickel steel tubing was examined at the well site by means of the Sonoscope. No further damage or outstandingly significant conditions were found. The tubing was remarkably free of general corrosion and, in fact, had the appearance of new, unused pipe.

Chemical and mechanical properties of the steel in both failures are listed in Table I; they are normal in all respects. The tubing had been heat-treated (normalized, 1575 to 1600 degrees F and tempered at 950 to 1050 degrees F) to develop a required 90,000 psi minimum yield strength and 25 percent minimum elongation (30 percent desired).

Since the steel was not defective in any obvious respect, the unusual appearance of fractures (both ductile and nonductile) suggested the possibility that the failures may have been induced by triaxial loading (resulting from tension weight of string), bursting stresses (contained gas), and complex stresses due to variable chilling by 50 degree F water. Calculations involving combination of all maximum known stresses established that a torsional load sufficient to cause a twist of 45 degrees in 30 feet, or one joint of pipe, would be required to develop a combined stress equal to the strength of the tubing. There was no conceivable source of such high twist, and it seems certain that the tubing failures were not purely mechanical.

It has been repeatedly suggested that the failures were caused by embrittlement, since the gas contained 10 percent hydrogen sulphide. The writer and his associates,<sup>1</sup> along with many other investigators, have shown that many steels exposed to hydrogen sulphide and water will lose considerable, if not all, ductility as a result of absorption of atomic hydrogen; but tensile and yield strengths remain essentially unchanged. In other words, steels embrittled by hydrogen do not fail under static tensile stresses until normal breaking loads are attained. Moreover, no



Figure 2—Other half of first failure.

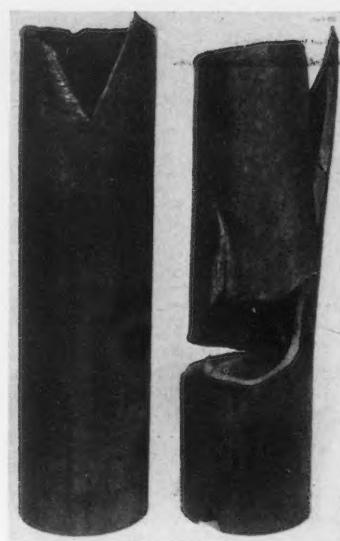


Figure 3—Both ends of second failure occurred at 1201 feet from surface.

significant change in ordinary mechanical properties was exhibited in tensile tests made on specimens of the tubing failures that had been immersed for short-time intervals in tap water saturated with hydrogen sulphide and carbon dioxide, and in well brine saturated with well gas.

It should not be overlooked, however, that absorption of atomic hydrogen may be a source of failure of steel under sulphide conditions by a process comparable to the well known blistering that occurs when some steels are acid pickled. Accumulation of molecular hydrogen at barriers within the steel (lattice imperfections, voids, inclusions, and sub-microscopic cracks) will produce high biaxial and triaxial stresses. These, plus normal service stresses, could conceivably induce brittle fracturing. There was no positive evidence that this had occurred in the 9 percent nickel steel tubing failures in the Marr well.

Since there was no possibility of fatigue, virtually the only remaining potential source of the tubing failures seemed to be stress-corrosion cracking. Stress-corrosion cracking is defined as spontaneous failure of metals by cracking under the combined action of corrosion and stress; the stress may be residual, applied, or both. The exact mechanism of the phenomenon is debatable. At least five theories<sup>2</sup> have been advanced, but it is suspected that no one theory applies in all cases of stress-corrosion cracking. It has been said that all metals can be made to fail by this process under some set of circumstances. Stress-corrosion cracking of metals is most common when the total stress is close to, or in, the plastic or yielding range.

### Duplication of Failures

Initial attempts to duplicate the service failures were made on specimens cut from the second tubing failure (yield strength, 121,000 pounds per square inch). Since oil well tubing is very often cold-straight-

TABLE I—Chemical and Mechanical Properties of Failed Tubing

	First Failure	Second Failure
<b>Analysis—</b>		
Carbon.....	0.095 percent	0.094 percent
Manganese.....	0.300 "	0.360 "
Phosphorus.....	0.025 "	0.035 "
Sulphur.....	0.027 "	0.026 "
Silicon.....	0.230 "	0.210 "
Copper.....	0.210 "	0.230 "
Nickel.....	8.910 "	8.990 "
Chromium.....	0.070 "	0.110 "
<b>Mechanical Properties—</b>		
Yield point (psi).....	101 600	121 300
Tensile strength (psi).....	121 000	132 300
Elongation (percent in 2 in.).....	28.0	28.0
Brinell hardness.....	250	260

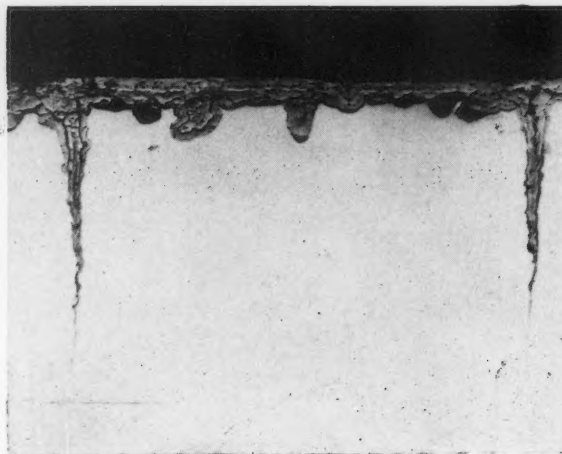


Figure 4—Stress corrosion cracks in 9 percent nickel steel beam specimen (fiber stress 70,000 psi) after 306 days in corrodent. (Unetched).

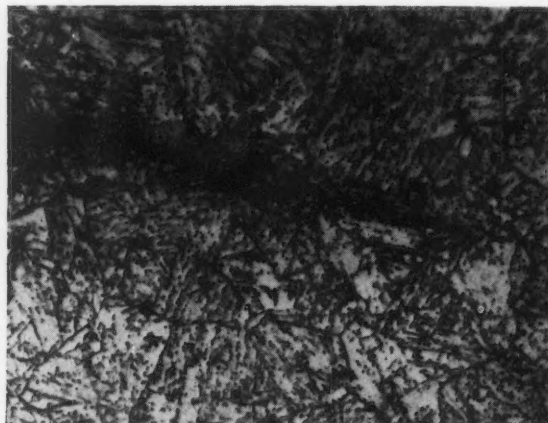


Figure 5—Portion of one of cracks in Figure 4. It is predominantly transgranular. Nital. X 1000.

ened (plastically deformed) during fabrication, these specimens were loaded above the yield strength as single point beams and were then submerged in tap water containing hydrogen sulphide and carbon dioxide in a test cell at 5000 pounds per square inch and 190 degrees F (bottom-hole conditions). Partial pressures of hydrogen sulphide and carbon dioxide were adjusted to approximate and maintain well fluid concentrations. The test period was six days (same as initial production test time), at the end of which time all specimens had failed, with brittle fractures in the zones of maximum stress (deformed regions).

Additional specimens from the second tubing failure were then single point beam loaded below the yield strength to maximum fiber stresses of 70,000, 90,000, and 110,000 pounds per square inch and immersed in tap water saturated with hydrogen sulphide and carbon dioxide at atmospheric pressure and 90 to 100 degrees F. The specimen loaded to 90,000 pounds per square inch failed in 14 days at a point where the applied stress was only 63,000 pounds per square inch; that loaded to 110,000 pounds per square inch failed in 70 days where the applied stress was only 36,000 pounds per square inch. The specimen at 70,-

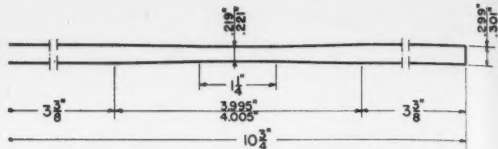


Figure 6—Tensile specimen for pre-stressed corrosion cracking test.

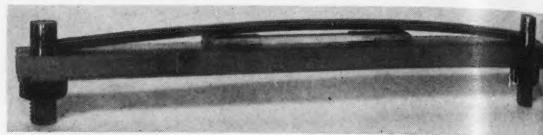


Figure 7—Method of applying bending load in stress-corrosion cracking tests.

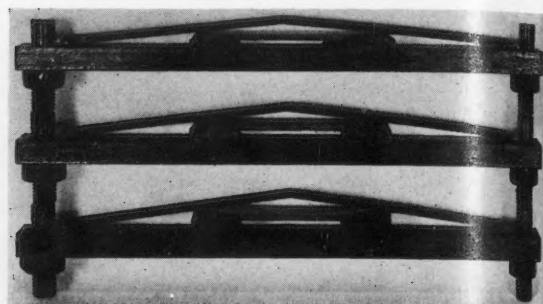


Figure 8—9 percent nickel steel stress-corrosion cracking failures.

000 pounds per square inch ultimately failed after 306 days at a point where the stress was 30,000 pounds per square inch. Figure 4 is a photomicrograph of the longitudinal section adjoining the complete fracture of this specimen, showing two stress-corrosion cracks. Figure 5 is a portion of one of these cracks at higher magnification and after etching the crack is predominantly transgranular. These erratic results suggested that failure had occurred in zones that had been stressed previously above the yield point by cold straightening the pipe from which the specimens were prepared.

A series of tests on 9 percent nickel and several other potentially usable alloys available as tubes, as well as API J-55 and N-80, was then undertaken to establish effect of pre-stressing above the yield point on susceptibility to stress-corrosion cracking. All alloys were re-heat treated to eliminate prior internal stresses. For the initial screening tests, specimens illustrated in Figure 6 were prestressed by loading in a tensile machine until the 1 1/4-inch length of uniform section in the center was stressed 1000 pounds per square inch above the yield strength determined by stress-strain diagrams. The rectangular section of the specimen is graduated so that pre-stressing at the ends of the 4-inch mid-zone is only two-thirds of the yield strength. After pre-stressing, the specimens were ground full length to a uniform section (0.180 inch by 0.200 inch) and 2-point beam loaded in a fixture shown in Figure 7. The applied fiber stress is constant between the two points of loading 4 inches apart. Hence, the variably prestressed zone is subjected to uniform applied stress. A few tests were also made on externally loaded specimens that were



re-heat treated but not pre-stressed. All specimens were immersed in tap water saturated with hydrogen sulphide and carbon dioxide at atmospheric pressure and room temperature. Additional laboratory and field tests are in progress on specimens of several steels pre-stressed midway between the yield and tensile strengths and to the ultimate strength of each metal.

### Laboratory Test Results

The more significant results for 9 percent nickel steel are summarized in Table II. With one exception (Test 7), all specimens of this steel failed sooner or later, depending primarily on the amount of pre-stressing above the yield strength and the magnitude of the applied fiber stress. Increasing hardness of the steel above Rockwell C24-26 and removal of corrosion products also appear to bear importantly on the exposure time to failure, which varied from seven hours to 202 days. Most of the failures, typical of which are those shown in Figure 8, were predominantly intergranular, with little or no evidence of pitting. Numerous small cracks parallel to the sides of the specimen, originating at and perpendicular to the main fracture surfaces (Figure 9), were present in many of the specimens. Short, longitudinal cracks were also found on top of some specimens (in the plane of maximum bending stresses); these, in association with the other cracks and the complete fracture, suggest a three-dimensional failure process.

The pre-stressed specimen in Test 2, Table II (applied fiber stress 60,000 pounds per square inch) was removed from the test cell for examination after 66 days of immersion. Mechanical cleaning of a tightly adherent film of corrosion products exposed numerous very fine cracks. When this specimen was replaced in the test cell, failure occurred in 24 hours after a total immersion time of 67 days.

A non-pre-stressed beam specimen (116,000 pounds per square inch yield strength) loaded to 60,000 pounds per square inch fiber stress was removed from the test cell after 115 days continuous immersion without cleaning. Beneath a tightly adherent film of corrosion products numerous barely visible cracks were found at the base of etched lines or 'striae'. A microscopic examination showed that these etched lines were elongated pits approximately 0.001-inch deep with cracks at the base penetrating 0.001 inch or less. Figure 10 is typical of the condition found. The cracks are too shallow to be certain, but they appear to be transgranular for the most part.

The foregoing indicates that prestressing beyond the yield strength greatly increases the susceptibility of 9 percent nickel steel tubing (as ordinarily heat treated) to failure in the selected sulphide environment (Tests 1, 3 and 5). Plastic deformation is not essential to the failure process, however (Test 4). A film of corrosion products appears to afford partial protection to the steel in either condition when the applied stress is relatively low (Tests 2, 4 and 6); but after the film is mechanically removed or ruptured (for example, by running a bottom-hole pressure gauge), failure may follow with disconcerting rapidity (Test 2). Also, it appears that the 'critical damaging

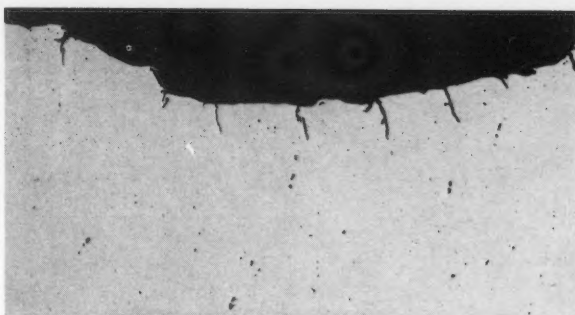


Figure 9—Small cracks originating at and perpendicular to main fracture faces.

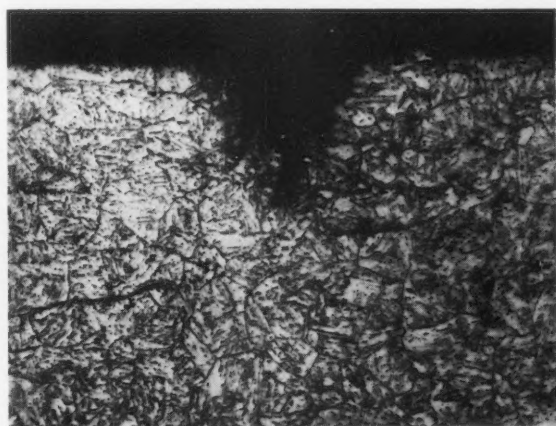


Figure 10—Cracks starting at pits in stress-corrosion cracking specimen. Note small stringers of non-metallic inclusions. Nital etch. X 500.

stress\* is lower than 60,000 pounds per square inch for 9 percent nickel steel as ordinarily processed for oil-well tubing, since all such specimens loaded to 60,000 pounds per square inch or more eventually failed.

Significant results of sulphide corrosion cracking tests on eleven other steels and three non-ferrous alloys are summarized in Table III. Thus far, under laboratory test conditions, failures have been induced in a 5 percent nickel-1 percent chromium steel (Tests 10 and 11); 5 percent nickel-molybdenum steel (Test 15); and Types 322 and 410 stainless steels (Tests 21,

\* "Critical damaging stress" is a relative term used for convenience; values for it cannot be employed for engineering purposes as they will vary with degree of plastic deformation of steel and corrosive environment.

TABLE II—Summary of Laboratory Sulphide Corrosion Cracking Test Results

9% Ni steel. Corrodent in all cases tap water saturated with H<sub>2</sub>S and CO<sub>2</sub> at atmospheric pressure and room temperature.

Test No.	Yield Strength (psi)	Hardness R <sub>c</sub>	Pre-Stressed <sup>1</sup>	Applied Fiber Stress (psi)	Time to Failure (Days)	Cleanings (No. of)
1	121,300	28.0	Yes	120,000	1½ to 4	0
2	121,300	28.0	Yes	60,000	67	1
3	116,000	27.5	No	120,000	31 hours	0
4	116,000	27.5	No	70,000	202	3
5	116,400	27.0	Yes <sup>2</sup>	110,000	7 hours	0
6	92,000	24.0	Yes	90,000	202	4
7	75,000	20.0	Yes	73,000	(370) <sup>3</sup>	23

<sup>1</sup> Pre-stressed 1,000 psi above determined yield strength.

<sup>2</sup> Pre-stressed to ultimate strength (132,000 psi).

<sup>3</sup> No failure; still under test.

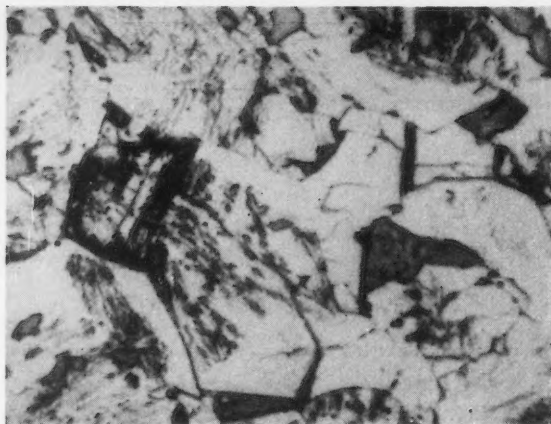


Figure 11—9 percent nickel steel structures after annealing followed by tempering at 800 degrees F. Hardness of dark constituent is 500-550 Brinell. Nital etch. X 2000.

22 and 23). Apparently 5 percent nickel-1 percent chromium steel is less susceptible to failure than 9 percent nickel steel with comparable mechanical properties. Test 12 (no failure in 267 days) indicates that the 'critical damaging stress' for the former steel is at least 70,000 pounds per square inch; Test 4 on non-pre-stressed 9 percent nickel steel resulted in a failure at 70,000 pounds per square inch applied fiber stress in 202 days.

The sensitizing effect of increasing amounts of plastic deformation is evident in the tests on 5 percent nickel-0.25 percent molybdenum steel under the same applied fiber stress (80,000 pounds per square inch). A specimen of this steel prestressed 1000 pounds per square inch above the yield strength (Test 13) has not failed in 266 days, but pre-stressing to the tensile strength (Test 15) resulted in a failure in 23 days. A specimen pre-stressed midway between the yield and tensile strengths (Test 14) is intact after 57 days. The 'critical damaging stress' for this steel is apparently above 80,000 pounds per square inch.

Despite pre-stressing to its tensile strength, API Grade N-80 steel (average mechanical properties) has not failed in 236 days under 90,000 pounds per

square inch applied fiber stress. Although pre-stressed only 1000 pounds per square inch above the yield strength, a specimen of 9 percent nickel steel with comparable yield strength (Test 6) failed at the same applied stress in 202 days.

The non-ferrous alloys tested have not shown any tendency to crack even when both hardness and applied fiber stress were high (Test 26).

With one exception (Test 15), all of the failures under laboratory test conditions have occurred in steels that had been heat treated to develop relatively high mechanical properties. This suggests that susceptibility to failure may be associated with the metallographic structure of the steel, and, therefore, might be minimized or eliminated by proper heat treating.

### Heat Treating and Metallographic Studies

After considering the heat treating and metallographic work on 9 percent nickel steel by Brophy and Miller,<sup>3</sup> it was postulated that susceptibility of 9 percent nickel steel to failure under hydrogen sulphide conditions might be due to untempered martensite resulting from decomposition of retained austenite upon cooling after tempering, or when the steel is plastically deformed. Untempered martensite is highly strained and, therefore, might be anodic to adjacent metal. If the untempered martensite were present as a more or less continuous network, it could be a source of stress-corrosion cracking.

A series of heat treating experiments and metallographic studies on 9 percent nickel steel were undertaken in an attempt to prove the theory. Figure 11 is the etched structure at 2000 diameters magnification of 9 percent nickel steel after annealing, followed by tempering for half an hour at 800 degrees F. The areas of dark grey constituent were found to have a microhardness equivalent to 500-550 Brinell, indicating that the constituent is probably martensite. Figure 12 represents the structure near the first failure in the 9 percent nickel steel tubing. The dark grey constituent is present at some of the grain boundaries. Appropriate etching has proved that this constituent is not a carbide.

Only one heat treatment was found which appears to eliminate the dark etching constituent in, and the susceptibility to sulphide corrosion cracking of, 9 percent nickel steel. The structure of this steel after double normalization at 1650 degrees and 1450 degrees F, followed by tempering at 1100 degrees F for two hours, is shown in Figure 13.

A pre-stressed beam specimen of 9 percent nickel steel so heat-treated has been immersed in the laboratory corrodant for 370 days (Test 7, Table II), during which time products of corrosion were removed 23 times. Thus far, there are no indications that sulphide corrosion cracking is occurring. Unfortunately,

TABLE III—Summary of Laboratory Sulphide Corrosion Cracking Test Results  
Corrodant: Tap water saturated with H<sub>2</sub>S and CO<sub>2</sub> at atmospheric conditions.

ALLOY	Test No.	Yield Strength (psi)	Hardness R <sub>c</sub>	Prestress (psi)	Applied Fiber Stress (psi)	Total Exposure (Days)	Cleanings (No. of)
9% Ni+0.30 Ti Steel...	8	99,700	22.0	100,700	96,000	468	22
5% Ni Steel...	9	92,000	15.0	63,000	60,000	521	21
5% Ni+1% Cr Steel...	10	113,000	29.0	114,000	110,000	(3) <sup>1</sup>	0
	11	113,000	29.0	114,000	90,000	(3) <sup>1</sup>	0
	12	113,000	29.0	114,000	70,000	267	20
5% Ni+0.25% Mo. Steel	13	82,300	24.0	83,300	80,000	266	19
	14	81,700	24.0	101,400	80,000	57	7
	15	81,700	24.0	124,200	80,000	(23) <sup>1</sup>	3
9% Cr+1.0% Mo. Steel.	16	99,000	25.0	100,000	90,000	524	22
5% Cr+0.55% Mo. Steel	17	92,000	26.0	93,000	90,000	524	23
API Grade J-55.....	18	59,400	15.0	60,400	58,000	538	22
API Grade N-80.....	19	95,000	24.0	96,000	90,000	525	22
	20	95,000	24.0	118,000	90,000	236	18
Stainless, 322.....	22	129,000	44.0	Not pre-stressed	120,000	(1) <sup>1</sup>	0
	21	129,000	44.0	1000 psi above yield strength	120,000	(5) <sup>1</sup>	1
Stainless 410.....	23	105,000	25.0	"	100,000	(123) <sup>1</sup>	17
Stainless 416.....	24	90,000	24.0	"	85,000	179	24
Monel.....	25	105,000	24.0	"	100,000	179	24
K Monel.....	26	120,000	37.0	"	115,000	179	24
Inconel.....	27	40,000	80.0R <sub>b</sub>	"	38,000	179	24

<sup>1</sup> Days to failure.

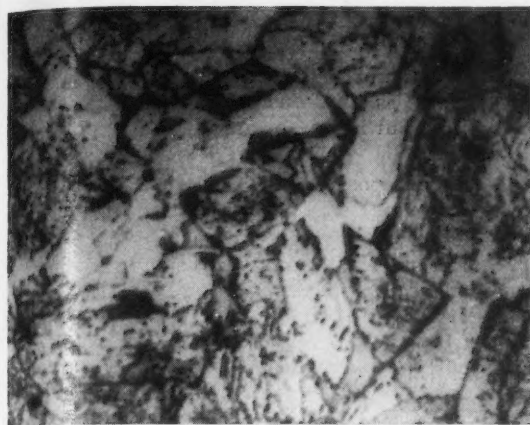


Figure 12—Structure near first tubing failure showing dark constituent at some grain boundaries. Nital etch. X 2000.

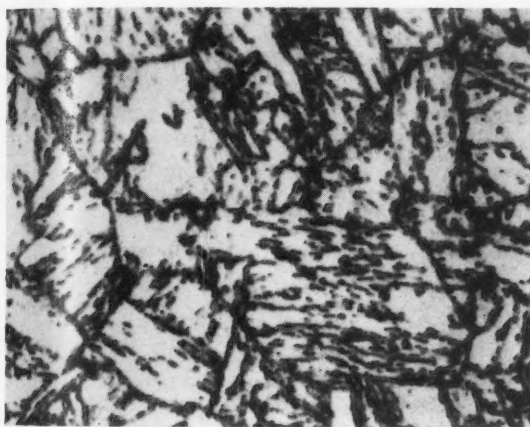


Figure 13—9 percent nickel steel double normalized 1650-1450 degrees F, tempered two hours at 1100 degrees F. Dark etching constituent is absent. Nital etch.

the successful heat treatment is impracticable from a tubing fabrication standpoint and the mechanical properties of the steel are undesirably low for high-pressure well service.

The effects of alloy 'stabilizing' additions have been explored only to a very limited extent, but there are indications that titanium and possibly molybdenum may be beneficial in reducing the sensitivity of some steels to the cracking phenomenon. It will be noted in Table III (Test 8) that a pre-stressed beam specimen of 9 percent nickel steel with 0.30 percent titanium has been under test for 468 days without failure under an applied fiber stress of 96,000 pounds per square inch. The structure of this steel is shown in Figure 14. Carbides are present but the dark etching constituent is absent. It seems probable that the titanium combines with the carbon of the steel to form titanium carbide, thereby minimizing or preventing austenite retention and thus avoiding the possibility of the subsequent formation of untempered martensite. Possibly, molybdenum in relatively large amount will serve in the same capacity. This appears to be indicated by the resistance of 9 percent chro-

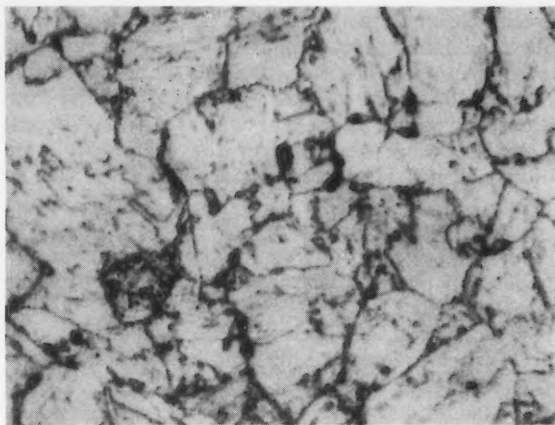


Figure 14—Structure of 9 percent nickel steel with 0.300 percent titanium. Dark constituent is absent. Nital etch.



Figure 15—9 percent nickel steel beam specimens after 272 days; tap water saturated with carbon dioxide. Specimen loaded up to 120,000 psi shows more corrosion in 4-inch high-stress zone.

mium-1.0 percent molybdenum steel to sulphite corrosion cracking (Test 16), in contrast with the failure of 12 percent chromium steel (Type 410) in 123 days (Test 23).

The absence of any known failure of 9 percent nickel steel tubing in sweet condensate service indicates that hydrogen sulphide is essential to the corrosion cracking process. This is confirmed in part by stress-corrosion tests on beam specimens from the second 9 percent nickel steel tubing failure in the Marr well. No failures have occurred after 454 days of immersion in tap water saturated with carbon dioxide, despite pre-stressing and beam loading to 100,000 and 120,000 pounds per square inch. Considerable corrosion in the 4-inch zone of high stress has occurred, as shown in Figure 15.

A comprehensive pilot production test was started at the Marr well in March, 1951. The programme, which is nearing completion, includes extensive materials studies under actual operating conditions, in which pre-stressed beam specimens, tube sections, and corrosion coupons of a large group of steels and non-ferrous alloys are being utilized. Detailed results of this work will be the subject of future reports. All of the failures of beam specimens that have occurred in the laboratory studies discussed herein have been duplicated under field conditions. In addition, failures have occurred in the field in beam specimens of 5



percent chrome steel, which so far has successfully withstood laboratory test conditions. This, and the fact that in some instances failures were more rapid in the field, indicates that the field operating conditions, involving higher pressures and different water, are more severe than the laboratory conditions selected for screening purposes.

### Summary

This investigation obviously is not completed, but the developments thus far seem to justify the following observations:

- (1) The failures of 9 percent nickel steel tubing at Pincher Creek were initiated by stress-corrosion cracking.
- (2) Susceptibility to failure in hydrogen sulphide environments is not limited to 9 percent nickel steel. There is evidence to indicate that most steels heat-treated to produce mechanical properties in or above a critical range represented approximately by Rockwell hardness C24 to C26 may be rendered susceptible to the failure process.
- (3) Plastic deformation greatly increases susceptibility of steel to the failure process, but it is not an essential condition if the protective film of corrosion products is ruptured or removed by some means, and the hardness of the steel is in or above the critical range Rockwell C24 to C26.
- (4) The mechanism of hydrogen sulphide corrosion cracking of steel has not been proved, but there

is evidence that a metallographic structural phase as a network may be the sensitizing factor.

- (5) Embrittlement of steel by exposure to hydrogen sulphide is not considered a primary cause of failure, but it may be an important contributory factor.
- (6) It may be possible to reduce susceptibility to the failure process by composition changes in steel.
- (7) There is no evidence thus far that 9 percent nickel steel will stress-corrosion crack in sweet condensate well service.

The author wishes to acknowledge the invaluable aid of his associates: A. J. Teplitz, C. N. Bowers, A. E. Wiehe, W. J. McGuire, J. K. Rodgers and R. R. Ellis.

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**Author's Note:** The paper above was presented as a progress report on a complex subject. A further progress report is presented by Messrs. Bowers, McGuire and Wiehe.

# Stress Corrosion Cracking of Steel Under Sulfide Conditions\*

By C. N. BOWERS, W. J. McGUIRE and A. E. WIEHE

## Introduction

**R**APID FAILURE of steel under hydrogen sulfide conditions has long been a serious problem to the materials engineer. The problem reached a new level of complexity with the development of the sour condensate Pincher Creek Field in Southwestern Alberta, Canada. During the production tests in the discovery well, rapid failures of API tubing, stainless steel wire lines and alloy steel fishing tools occurred. When the second well in this field, the Walter Marr No. 1, was completed 9 percent nickel steel tubing was installed. This selection was dictated by the fact that this grade of tubing has performed with excellent results in many sweet condensate wells in Mississippi, Louisiana and Texas, plus the fact that other nickel alloys have given outstanding performance in oil field service where sulfide corrosion conditions existed.

Production in the Marr well comes from the Madison lime at the depth interval approximately 12,200-12,750 feet. The gas-oil ratio is about 26,000 and the gas contains 10.2 percent hydrogen sulfide and 6.3 percent carbon dioxide by volume. During six days of initial testing, the well was produced at an average rate of 7.5 million cubic feet per day, yielding approximately 300 barrels per day of distillate along with about the same volume of formation water. This water, containing some 37,500 ppm dissolved solids, was high in bicarbonates and, of course, hydrogen sulfide. The bottom hole pressure and temperature were 4868 psi and 188 degrees F, respectively, at 12,200 feet. The wellhead pressure shut in was 3236 psig and at 7.5 million cubic feet per day flow rate averaged 1350 psig.

Two very unexpected failures in the 9 percent nickel steel tubing after only six days of service during production tests led to an extensive laboratory and field investigation to determine the cause of and the remedy for these failures. A previous paper<sup>1</sup> on this subject gave the history and analysis of the failures and tentatively concluded that the failures were due to stress-corrosion cracking. Embrittlement resulting from exposure to hydrogen sulfide was not considered to be a primary cause of failure, but may have been an important contributory factor. The present paper is a progress report of laboratory work on this problem.

## Preliminary Laboratory Tests

In most of the initial screening tests specimens

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## Abstract

Rapid failures of tubing in a sour condensate well led to an extensive laboratory and field investigation to determine the cause and remedy for these failures. Apparently, failures occur because of stress corrosion cracking. Embrittlement resulting from exposure to moist hydrogen sulfide is not considered a primary cause of failures, but it may have been an important contributing factor.

The exact mechanism of failure has not yet been definitely established. The failure process can be prevented or appreciably retarded by suitable heat treatment and/or change in steel composition. Susceptibility to failure of most steels is increased by plastic deformation.

In most instances failures were more rapid in field tests, which indicated that the field operating conditions involving pressure, flow, and formation water were more severe than the laboratory test conditions selected. In both tests there was little if any advantage for alloy steels over plain carbon steels. It was apparent, however, that for most steels, limitations on the maximum permissible mechanical properties are just as important and necessary as minimum limits.

\*A paper presented at the Eighth Annual Conference, National Association of Corrosion Engineers, Galveston, Texas, March 10-14, 1952.

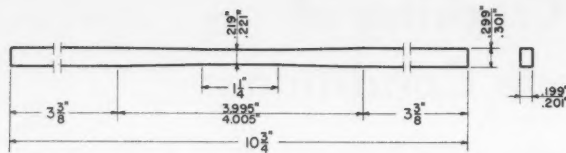


Figure 1—Tensile specimen for pre-stressed corrosion cracking test.



Figure 2—Method of applying load in stress-corrosion cracking test.

TABLE I—Summary of Preliminary Laboratory Sulfide Corrosion Cracking Test Results<sup>1</sup>

ALLOY	Test No.	Yield Strength (psi)	Hardness R <sub>c</sub>	Pre-stress (psi)	Applied Fiber Stress (psi)	Time to Failure (Days)	Total Exposure (Days)	No. of Cleanings	No. of Days From Last Cleaning to Failure
9% Ni Steel.....	1	121,300	28.0	122,300	120,000	1 1/2-4	.....	0	.....
	2	121,300	28.0	122,300	60,000	67	.....	1	1
	3	116,000	27.5	None	120,000	31 hours	.....	0	.....
	4	116,000	27.5	None	70,000	202	.....	3	2
	5	116,400	27.0	132,000	110,000	7 hours	.....	0	.....
	6	92,000	24.0	93,000	90,000	202	.....	4	7
	7	75,000	20.0	76,000	73,000	.....	497	41	.....
9% Ni+0.30% Ti Steel.....	8	99,700	22.0	100,700	96,400	.....	595	40	.....
	9	62,000	15.0	63,000	60,000	.....	662	39	.....
5% Ni+0.25% Mo Steel.....	10	82,300	24.0	83,300	80,000	.....	393	37	.....
	11	81,700	24.0	101,400	80,000	.....	184	25	.....
	12	81,700	24.0	124,200	80,000	23	.....	3	3
5% Ni+1.0% Cr Steel.....	13	113,000	29.0	114,000	110,000	3	.....	0	.....
	14	113,000	29.0	114,000	90,000	3	.....	0	.....
	15	113,000	29.0	114,000	70,000	.....	394	38	.....
5% Cr+0.55% Mo Steel.....	16	92,000	26.0	93,000	90,000	.....	661	41	.....
	17	99,000	25.0	100,000	90,000	.....	661	41	.....
	18	59,400	15.0	60,400	58,000	.....	665	40	.....
API Grade N-80.....	19	95,000	24.0	96,000	90,000	.....	662	40	.....
	20	95,000	24.0	118,000	100,000	.....	363	36	.....
	21	95,000	24.0	118,000	90,000	.....	363	36	.....
Stainless 322.....	22	129,000	44.0	130,000	120,000	5	.....	1	1
	23	129,000	44.0	None	120,000	2	.....	0	.....
	24	135,000	45.0	136,000	108,000	0-16 hours	.....	0	.....
	25	135,000	46.0	136,000	54,000	9	.....	2	1
	26	135,000	46.0	136,000	35,700	.....	79	11	.....
Stainless 410.....	27	105,000	25.0	106,000	100,000	123	.....	17	7
	28	105,000	25.0	None	100,000	.....	134	20	.....
Stainless 416.....	29	90,000	24.0	91,000	85,000	.....	306	42	.....
	30	105,000	24.0	106,000	100,000	.....	306	42	.....
K Monel.....	31	120,000	37.0	121,000	115,000	.....	306	42	.....
Inconel.....	32	40,000	80.0R <sub>B</sub>	41,000	38,000	.....	306	42	.....

<sup>1</sup> Corrodent: Tap water saturated with H<sub>2</sub>S and CO<sub>2</sub> at atmospheric conditions.

TABLE I-A—Summary of Chemical Analyses and Mechanical Properties of Materials Included in Table I

ALLOY	Test Number	CHEMICAL ANALYSES								MECHANICAL PROPERTIES			Heat Treatment <sup>1</sup>
		C	Mn	Cr	Ni	Mo	Cu	Ti	Other	Yield Strength (psi)	Tensile Strength (psi)	Hardness R <sub>c</sub>	
9% Ni Steel.....	1, 2	0.11	0.42	.....	8.89	.....	0.35	.....	.....	121,300	.....	28.0	N 1575° F; T 950° F for 1/2 hr.; AC
	3, 4, 5	.....	.....	.....	.....	.....	.....	.....	.....	116,000	.....	27.5	N 1575° F; T 950° F for 1/2 hr.; FC
	6	.....	.....	.....	.....	.....	.....	.....	.....	92,000	.....	24.0	Double N 1650° F & 1450° F; T 1030° F for 2 hrs.; AC
	7	.....	.....	.....	.....	.....	.....	.....	.....	75,000	.....	20.0	Double N 1650° F & 1450° F; T 1100° F for 2 hrs.; AC
9% Ni+0.30% Ti Steel.....	8	0.04	0.40	0.51	9.10	.....	0.04	0.29	.....	99,700	111,000	22.0	N 1575° F; T 750° F for 1/2 hr.; AC
5% Ni Steel.....	9	0.10	0.47	0.18	4.94	0.06	0.17	.....	.....	62,000	97,750	15.0	N 1575° F; T 900° F for 1/2 hr.; AC
5% Ni+0.25% Mo Steel.....	10	0.22	0.46	0.06	4.83	0.24	.....	.....	.....	82,300	123,600	24.0	N 1575° F; FC
	11, 12	.....	.....	.....	.....	.....	.....	.....	.....	81,700	123,600	24.0	Double N 1650° F & 1450° F; T 1100° F for 2 hrs.; AC
5% Ni+1.0% Cr Steel.....	13, 14, 15	0.19	0.41	1.01	4.81	0.05	.....	.....	.....	113,000	133,000	29.0	N 1575° F; T 1050° F for 1/2 hr.; AC
5% Cr+0.55% Mo Steel.....	16	0.09	0.45	4.96	0.24	0.49	0.13	.....	.....	92,000	.....	26.0	N 1675° F; T 1150° F for 1/2 hr.; AC
9% Cr+1.0% Mo Steel.....	17	0.12	0.42	8.63	0.24	0.99	.....	.....	.....	99,000	.....	25.0	N 1700° F; T 1200° F for 1/2 hr.; AC
API Grade J-55.....	18	0.40	1.20	.....	.....	.....	.....	.....	.....	59,400	.....	15.0	HR; T 800° F for 1/2 hr.; FC
API Grade N-80.....	19, 20, 21	0.45	1.80	.....	.....	.....	.....	.....	.....	95,000	.....	24.0	N 1600° F; T 800° F for 1/2 hr.; FC
	22, 23	0.07	0.50	17.0	7.0	.....	.....	0.70	0.20 Al	129,000	.....	44.0	WQ 1900° F; T 950° F for 1/2 hr.; AC
Stainless 322.....	24, 25, 26	.....	.....	.....	.....	.....	.....	.....	.....	135,000	.....	46.0	WQ 1900° F; T 950° F for 1/2 hr.; AC
Stainless 410.....	27, 28	0.15 (max.)	1.00 (max.)	11.5 to 13.5	.....	.....	.....	1.00 Si	.....	105,000	.....	25.0	WQ 1750° F; T 1100° F for 1/2 hr.; AC
Stainless 416.....	29	0.15 (max.)	1.25 (max.)	12.0 to 14.0	.....	0.60	.....	1.18 to 1.35 S	.....	90,000	.....	24.0	WQ 1850° F; T 1100° F for 1/2 hr.; AC
		.....	.....	.....	.....	.....	.....	1.00 Si	.....	.....	.....	.....	.....
Monel.....	30	.....	1.0	.....	67.0	.....	30.0	1.5 Fe	.....	105,000	.....	24.0	HR
K Monel.....	31	.....	.....	.....	66.0	.....	29.0	2.75 Al	.....	120,000	.....	37.0	HR; Age 950° F for 10 hrs.; AC
Inconel.....	32	.....	.....	15.0	80.0	.....	.....	5.0 Fe	.....	40,000	.....	80.0R <sub>B</sub>	HR

<sup>1</sup> N=Normalized; T=Temper; AC=Air Cool; FC=Furnace Cool; WQ=Water Quench; HR=Hot Rolled.



Several significant facts were apparent from these results. The amount of prestressing beyond the yield strength and the magnitude of the applied stress influence the susceptibility to failure in the selected environment of some steels that have been heat treated to produce mechanical properties in or above a critical range represented approximately by Rockwell hardness C24 to C26 (Tests 1, 3, 5, 12 and 27). Prestressing is not, however, necessary (Test 4). There was no intended prestressing in Test 3, but the applied fiber stress exceeded the yield strength.

A film of corrosion products affords at least partial protection to steel with or without prestressing (Tests 2, 4, 6 and 12). After the film is mechanically removed, failure may occur quite rapidly (Test 2). Protection by corrosion products is, of course, undependable.

There were indications that the degree of plastic deformation increases the sensitivity of some steel to failure (Tests 10, 11 and 12). However, all steels are not sensitized as is shown by Test 21, which has not failed after 363 days though prestressed to the tensile strength. This does not hold in Test 20 because the specimen was loaded in bending sufficiently to exceed the yield strength, in which case Hooke's law does not apply. Consequently, in this case, the actual applied fiber stress was not the equivalent of the stress that would be present if the steel were still within the elastic range.

In susceptible steels, there is a "critical" stress, sometimes referred to by others as "threshold" stress below which failure will not occur (Tests 15 and 26). The values of this term are only relative and will vary with the steel, the degree of plastic deformation and the selected corrosive environment.

Apparently the susceptibility to the failure process of some steels can be eliminated or appreciably reduced by suitable heat treatment and/or change in the steel composition. The 9 percent nickel steel (Test 7) double normalized from 1650 degrees F and 1450 degrees F and tempered to 1100 degrees F for two hours has not failed after 497 days though prestressed 1000 psi above the yield strength and loaded in bending to a fiber stress almost equal to the yield strength. The addition of titanium, a carbide former, to 9 percent nickel steel (Test 8) has apparently prevented failure, since a specimen of this steel has not failed in 595 days. A specimen of straight 9 percent nickel steel (Test 6) failed after 202 days with a lower applied fiber stress.

TABLE II—Summary of Laboratory Sulfide Corrosion Cracking Test Results Showing Effects of Plastic Deformation of API Grades J-55 and N-80<sup>1</sup>

ALLOY	Test No.	Yield Strength (psi)	Hardness R <sub>c</sub>	Prestress (psi)	Applied Fiber Stress (psi)	Time to Failure (days)	Total Exposure (days)	No. of Cleanings	No. of Days From Last Cleaning to Failure
API Grade J-55.....	33	114,700	30.0	125,000	90,000	11	..	1	5
	34	115,800	31.0	125,000	80,000	12	..	1	6
	35	99,200	22.0	107,900	79,100	..	11	3	..
	36	99,100	24.0	107,900	69,200	..	11	3	..
	37	98,100	23.0	114,100	79,100	..	11	3	..
	38	99,300	23.0	115,200	69,200	..	11	3	..
API Grade N-80.....	39	93,200	28.0	117,000	73,600	0-16 hrs.	..	0	..
	40	95,200	28.0	118,800	64,400	..	81	11	..
	41	92,200	30.0	139,900	73,600	0-16 hrs.	..	0	1
	42	92,700	30.0	139,300	64,400	2-3	..	1	..
	43	95,600	24.0	107,000	76,200	..	11	3	..
	44	94,800	24.0	124,300	76,200	0-16 hrs.	..	0	..
API Grade N-80.....	45	98,700	28.0	116,800	78,400	..	79	11	..
	46	98,700	27.0	117,700	68,600	..	79	11	..
	47	98,600	28.0	135,300	78,400	76	..	11	2
	48	100,600	28.0	135,300	68,600	..	79	11	..
API Grade N-80.....	49	81,600	24.0	100,600	64,000	..	91	13	..
	50	83,000	21.0	103,200	56,000	..	91	13	..
	51	77,000	22.0	117,200	64,000	..	80	11	..
	52	81,400	21.0	123,300	56,000	..	80	11	..
	53	81,400	22.0	104,700	64,000	..	91	13	..
API Grade N-80.....	54	82,800	23.0	105,000	56,000	..	91	13	..
	55	82,800	23.0	127,700	64,000	..	91	13	..
	56	80,200	24.0	128,200	56,000	..	91	13	..
	57	89,900	21.0	105,300	68,800	..	80	11	..
	58	87,500	21.0	104,100	60,200	..	80	11	..
API Grade N-80.....	59	84,700	20.0	120,500	68,800	..	80	11	..
	60	83,500	21.0	122,600	60,200	..	80	11	..
	61	93,600	26.0	113,500	73,600	..	79	11	..
	62	93,200	27.0	113,000	64,400	..	79	11	..
	63	93,100	26.0	134,300	73,600	..	79	11	..
API Grade N-80.....	64	93,200	26.0	135,000	64,400	..	79	11	..
	65	77,900	22.0	95,900	62,400	..	56	8	..
	66	77,400	20.0	95,100	54,600	..	56	8	..
	67	78,200	21.0	112,800	62,400	..	56	8	..
	68	78,700	22.0	113,200	54,600	..	56	8	..
API Grade N-80.....	69	94,400	23.0	112,300	78,400	..	56	8	..
	70	101,200	25.0	119,000	68,600	..	56	8	..
	71	98,200	26.0	133,500	78,400	..	56	8	..
	72	97,500	25.0	132,600	68,600	..	49	7	..

<sup>1</sup> Corrodent: Tap water saturated with H<sub>2</sub>S and CO<sub>2</sub> at atmospheric conditions.

The limited number of nonferrous alloys tested (Tests 30-32, inclusive) have not shown cracking tendencies regardless of the hardness or the applied fiber stress.

The ordinary tubing steels (API Grades J-55 and N-80) were at least the equal of low alloy steels under the conditions of loading and environment utilized (Tests 18, 19, 20 and 21). In view of the results obtained plus the possible use of substitutes for Grade N-80 steel due to alloy shortages, a series of tests on regular grades of J-55 and N-80, Grade N-80 substitutes and some steels of proved susceptibility were made to determine the effect of plastic deformation resulting from the cold straightening that is necessary in the manufacture of pipe.

#### Effect of Plastic Deformation

The results of tests on several of the above materials, heat treated to various hardness levels and prestressed midway between the yield and tensile strengths and to the tensile strength and then loaded

TABLE II-A—Summary of Chemical Analyses and Mechanical Properties of Steels Included in Table II

ALLOY	Test No.	CHEMICAL ANALYSIS					MECHANICAL PROPERTIES			Heat Treatment <sup>1</sup>
		C	Mn	Cr	Mo	V	Yield Strength (psi)	Tensile Strength (psi)	Hardness R <sub>c</sub>	
API Grade J-55.....	33	0.40	1.20	.....	.....	.....	114,700	125,000	30.0	WQ 1550° F; T 900° F; AC
	34	.....	.....	.....	.....	.....	115,800	125,800	31.0	WQ 1550° F; T 900° F; AC
	35	.....	.....	.....	.....	.....	99,200	107,800	22.0	WQ 1550° F; T 1/2 hr. 1100° F; AC
	36	.....	.....	.....	.....	.....	99,100	107,800	24.0	WQ 1500° F; T 1/2 hr. 1100° F; AC
	37	.....	.....	.....	.....	.....	98,100	114,500	23.0	WQ 1500° F; T 1/2 hr. 1100° F; AC
	38	.....	.....	.....	.....	.....	99,300	114,500	23.0	WQ 1500° F; T 1/2 hr. 1100° F; AC
API Grade N-80.....	39	0.41	1.83	.....	0.22	.....	93,200	139,600	28.0	N 1575° F for 1/2 hr. (Lab.)
	40	.....	.....	.....	.....	.....	95,200	139,600	28.0	N 1575° F for 1/2 hr. (Lab.)
	41	.....	.....	.....	.....	.....	92,200	139,600	30.0	N 1575° F for 1/2 hr. (Lab.)
	42	.....	.....	.....	.....	.....	92,700	139,600	30.0	N 1575° F for 1/2 hr. (Lab.)
	43	.....	.....	.....	.....	.....	95,600	100,000	24.0	N 1600° F; T 800° F; FC
	44	.....	.....	.....	.....	.....	94,800	100,000	24.0	N 1600° F; T 800° F; FC
API Grade N-80 Substitute.....	45	0.40	1.65	.....	0.14	.....	98,700	135,300	28.0	N 1575° F for 1/2 hr.; AA (Lab.)
	46	.....	.....	.....	.....	.....	98,700	135,300	27.0	N 1575° F for 1/2 hr.; AA (Lab.)
	47	.....	.....	.....	.....	.....	98,600	135,300	28.0	N 1575° F for 1/2 hr.; AA (Lab.)
	48	.....	.....	.....	.....	.....	100,600	135,300	28.0	N 1575° F for 1/2 hr.; AA (Lab.)
API Grade N-80 Substitute.....	49	0.41	1.64	0.18	0.06	.....	81,600	120,200	24.0	N 1525° F for 2 hrs.; AC (Mill)
	50	.....	.....	.....	.....	.....	83,000	120,200	21.0	N 1525° F for 2 hrs.; AC (Mill)
	51	.....	.....	.....	.....	.....	77,000	120,200	22.0	N 1525° F for 2 hrs.; AC (Mill)
	52	.....	.....	.....	.....	.....	81,400	120,200	21.0	N 1525° F for 2 hrs.; AC (Mill)
API Grade N-80 Substitute.....	53	0.44	1.69	0.53	0.06	.....	81,400	127,900	22.0	N 1525° F for 2 hrs.; AC (Mill)
	54	.....	.....	.....	.....	.....	82,800	127,900	23.0	N 1525° F for 2 hrs.; AC (Mill)
	55	.....	.....	.....	.....	.....	82,800	127,900	23.0	N 1525° F for 2 hrs.; AC (Mill)
	56	.....	.....	.....	.....	.....	80,200	127,900	24.0	N 1525° F for 2 hrs.; AC (Mill)
API Grade N-80 Substitute.....	57	0.44	1.71	0.07	.....	0.13	89,900	121,500	21.0	N 1525° F for 2 hrs.; AC (Mill)
	58	.....	.....	.....	.....	.....	87,500	121,500	21.0	N 1525° F for 2 hrs.; AC (Mill)
	59	.....	.....	.....	.....	.....	84,700	121,500	20.0	N 1525° F for 2 hrs.; AC (Mill)
	60	.....	.....	.....	.....	.....	85,500	121,500	21.0	N 1525° F for 2 hrs.; AC (Mill)
API Grade N-80 Substitute.....	61	0.44	1.68	0.25	0.07	.....	93,600	133,600	26.0	Mill normalized
	62	.....	.....	.....	.....	.....	93,200	133,600	27.0	Mill normalized
	63	.....	.....	.....	.....	.....	93,100	133,600	26.0	Mill normalized
	64	.....	.....	.....	.....	.....	93,200	133,600	27.0	Mill normalized
API Grade N-80 Substitute.....	65	0.42	1.47	.....	.....	.....	77,900	112,500	22.0	Mill normalized
	66	.....	.....	.....	.....	.....	77,400	112,500	20.0	Mill normalized
	67	.....	.....	.....	.....	.....	78,200	112,500	21.0	Mill normalized
	68	.....	.....	.....	.....	.....	78,700	112,500	22.0	Mill normalized
API Grade N-80 Substitute.....	69	0.44	1.58	0.21	0.07	.....	94,400	133,000	23.0	Mill normalized
	70	.....	.....	.....	.....	.....	101,200	133,000	25.0	Mill normalized
	71	.....	.....	.....	.....	.....	98,200	133,000	26.0	Mill normalized
	72	.....	.....	.....	.....	.....	97,500	133,000	25.0	Mill normalized

<sup>1</sup> WQ=Water Quench; N=Normalized; T=Temper; AC=Air Cool; FC=Furnace Cool; AA=Agitated Air (Fan Cool).

in bending to 80 percent and 70 percent of the yield strength, are summarized in Tables II and III. The compositions of the steels and their heat treatments are given in Tables II A and III A.

Ordinary API Grade J-55 steel prestressed to 1000 psi above the yield strength and loaded to a fiber stress of 48,000 psi has not failed after 665 days (Test 18). This same grade of steel failed in 11 and 12 days (Tests 33 and 34) after being heat treated to Rockwell hardness C30 and C31 and loaded to a fiber stress of 90,000 and 80,000 psi, respectively. It is possible that failure in these tests may have been due primarily to the higher applied stresses. Specimens of the same steel treated to Rockwell C23 to C24 have not been under test long enough for results to be of real significance at this time.

Grade N-80 steel, in the normalized condition with higher than normal manganese content (Tests 39 to 44, inclusive) is affected both by the degree of plastic deformation and the magnitude of the applied stress. It is significant that Test 40 is still intact after 81 days, though the hardness is Rockwell C28. The applied stress was 70 percent of the yield strength and the specimen was prestressed midway between the yield and tensile strengths. With an applied stress of 80 percent of the yield strength and the same prestressing failure occurred within 16 hours (Test 39). At the hardness level of Rockwell C30, failures occurred within 16 hours and in from 2 to 3 days when fiber stresses of 80 percent and 70 percent, respec-

tively, were applied to specimens prestressed to the tensile strength (Tests 41 and 42). The application of fiber stress of 80 percent of the yield strength to the same steel tempered at 1000 degrees F to reduce the hardness to Rockwell C24 resulted in failure within 16 hours when the specimen was prestressed to the tensile strength, whereas failure has not occurred after 11 days when the specimen was prestressed midway between the yield and tensile strengths (Tests 44 and 43).

In another group of regular Grade N-80 specimens (Tests 45 to 48, inclusive) the one prestressed to the tensile strength and loaded to a fiber stress of 80 percent of the yield strength failed in 76 days (Test 47). The other specimens of this group have been on test for only 79 days and failure may still occur. The steel in these tests was made by a different manufacturer than that of Tests 39 to 42, inclusive. The chemical composition and mechanical properties were essentially the same, except that the yield strength of the steel in Tests 45 to 48, inclusive, was slightly higher. Despite this, there was a wide difference in the results obtained. Also, specimens of one substitute Grade N-80 steel (Tests 61 to 64, inclusive) under the same prestress and load conditions of Tests 39 to 42, inclusive, but with a hardness of Rockwell C26 to C27, have not failed in 79 days. Further, another group of substitute Grade N-80 specimens (Tests 69 to 72, inclusive) with still slightly lower hardness and tensile strength, but

with higher yield strength than that of Tests 39 to 42, inclusive, is still intact after 49 to 56 days, though prestressed to the same degree and loaded with slightly higher applied stresses than those in Tests 39 to 42, inclusive. None of the other substitute Grade N-80 steels with lower mechanical properties have failed (Tests 49 to 60, inclusive and Tests 65 to 68, inclusive).

While the tests in Table II have not been in progress long enough to be definitely conclusive, it is apparent that the hardness of the steel as ordinarily determined is not a precise criterion of susceptibility. It is also indicated that the other mechanical properties are similarly deficient. The results do suggest that the failure process is associated with metallographic structural characteristics of the steel.

The tests on prestressed nickel steels with various additions of other alloying elements (Table III) show most of the steels to be susceptible to early

failure in the higher hardness range when subjected to high applied stress and severe plastic deformation. This sensitivity has also been shown by some of these steels at lower hardness levels (Tests 82, 89, 90, 91 and 92). While the tests have been of short duration only, it is indicated that lowering the hardness and other properties lessens the sensitivity to the failure process. It again appears that susceptible materials have a "critical" stress from a comparison of Test 82 with 84, both prestressed to the tensile strength but loaded to a fiber stress of 70,800 and 61,900 psi, respectively.

### Metallographic Studies

During the early stages of the investigation, it was theorized from the work of Brophy and Miller,<sup>2</sup> that failure of 9 percent nickel steel might be due to untempered martensite resulting from decomposition of retained austenite upon cooling after tempering, or during plastic deformation. Untempered martensite is highly strained and, therefore, might very well be anodic to adjacent metal. If this constituent were present as a continuous network or phase, it could be a source of stress-corrosion cracking.

Examination of the actual tubing failures and the laboratory test specimens of 9 percent nickel steel showed the presence, in the grain boundaries, of a dark etching constituent believed to be martensite. Figure 3 is a representation of this condition. This dark etching constituent was successfully eliminated (Figure 4) by double normalizing at 1650 degrees and 1450 degrees F followed by tempering at 1100 degrees F for two hours. A prestressed beam of 9 percent nickel steel so treated is still intact after 497 days

TABLE III—Summary of Laboratory Sulfide Corrosion Cracking Test Results Showing Effect of Plastic Deformation on Low Alloy Steels<sup>1</sup>

ALLOY	Test No.	Yield Strength (psi)	Hardness R <sub>c</sub>	Pre-stress (psi)	Applied Fiber Stress (psi)	Time to Failure (days)	Total Exposure (days)	No. of Cleanings	No. of Days From Last Cleaning to Failure
3% Ni+0.26% Mo+0.38% Cu Steel..	73	92,000	30.0	133,500	90,000	5	..	2	1
	74	95,200	27.0	126,000	90,000	1/2-3	..	0	..
	75	120,000	29.0	150,000	100,000	1/2-3	..	0	..
3% Ni+0.20% Mo+0.10% Cu+0.08% Al Steel....	76	96,600	28.0	123,000	90,000	1/2-3	..	0	..
	77	93,500	25.0	116,000	90,000	5	..	2	1
	78	88,400	20.0	110,000	80,000	..	175	26	..
3% Ni+1.0% Cr Steel.....	79	114,200	26.0	120,900	110,000	39	..	5	1
	80	114,800	26.0	125,600	110,000	18-21	..	2	4
	81	90,100	22.0	104,100	70,800	..	11	4	..
	82	87,200	23.0	112,100	70,800	3-4	..	3	1
	83	88,300	22.0	102,800	61,900	..	11	4	..
3% Ni+0.46% Mo Steel.....	84	88,400	23.0	113,600	61,900	..	11	4	..
	85	112,900	28.0	124,000	91,200	0-1	..	0	..
	86	115,600	29.0	135,400	91,200	0-1	..	0	..
	87	93,800	23.0	103,200	75,600	41	..	6	3
9% Ni Steel.....	88	94,200	22.0	111,800	75,600	..	49	7	..
	89	98,000	20.0	105,600	76,800	2	..	2	..
	90	94,700	20.0	106,900	67,200	11	..	2	1
	91	94,300	20.0	108,800	76,800	1-2	..	1	..
9% Ni Steel.....	92	97,200	21.0	109,300	67,200	1-2	..	1	..

<sup>1</sup> Corrosion: Tap water saturated with H<sub>2</sub>S and CO<sub>2</sub> at atmospheric conditions.

TABLE III-A—Summary of Chemical Analyses and Mechanical Properties of Steels Included in Table III

ALLOY	Test No.	CHEMICAL ANALYSES								MECHANICAL PROPERTIES			Heat Treatment <sup>1</sup>
		C	Mn	Cr	Ni	Mo	Cu	Ti	Other	Yield Strength (psi)	Tensile Strength (psi)	Hardness R <sub>c</sub>	
3% Ni+0.26% Mo+0.38% Cu Steel.....	73	0.22	0.78	0.01	4.89	0.26	0.38	....	....	92,000	133,500	30.0	N 1600° F; T 1025° F; AC
	74	...	...	...	...	...	...	...	...	95,200	126,000	27.0	N 1600° F; T 1100° F; AC
	75	...	...	...	...	...	...	...	...	120,000	150,000	29.0	N 1600° F; T 1175° F; T 1250° F; AC
3% Ni+0.20% Mo+0.10% Cu+0.08% Al Steel....	76	0.15	0.81	....	5.10	0.20	0.10	....	0.08 Al	96,600	123,000	28.0	N 1600° F; T 950° F; AC
	77	...	...	...	...	...	...	...	...	93,500	116,000	25.0	N 1600° F; T 1025° F; AC
	78	...	...	...	...	...	...	...	...	88,400	110,000	20.0	N 1600° F; T 1100° F; AC
3% Ni+1.0% Cr Steel..	79	0.19	0.41	1.01	4.81	0.05	....	....	....	114,200	125,600	26.0	N 1575° F; T 1000° F; AC
	80	...	...	...	...	...	...	...	...	114,800	125,600	26.0	N 1575° F; T 1000° F; AC
	81	...	...	...	...	...	...	...	...	90,100	112,100	22.0	N 1575° F; T 1100° F; AC
	82	...	...	...	...	...	...	...	...	87,200	112,100	23.0	N 1575° F; T 1100° F; AC
	83	...	...	...	...	...	...	...	...	88,300	113,600	22.0	N 1575° F; T 1100° F; AC
3% Ni+0.46% Mo Steel.....	84	...	...	...	...	...	...	...	...	88,400	113,600	23.0	N 1575° F; T 1100° F; AC
	85	0.18	0.67	0.01	5.33	0.46	....	....	....	112,900	135,000	28.0	N 1575° F; T 1000° F; AC
	86	...	...	...	...	...	...	...	...	115,600	135,400	29.0	N 1575° F; T 1000° F; AC
	87	...	...	...	...	...	...	...	...	93,800	114,000	23.0	N 1575° F; T 1100° F; for 3 hrs.; T 1130° F for 1 hr.; AC
9% Ni Steel.....	88	...	...	...	...	...	...	...	...	94,200	111,800	22.0	N 1575° F; T 1100° F for 3 hrs. T 1130° F for 1 hr.; AC
	89	0.11	0.42	....	8.89	....	0.35	....	....	98,000	108,000	20.0	N 1575° F; T 1000° F; AC
	90	...	...	...	...	...	...	...	...	94,700	108,500	20.0	N 1575° F; T 1000° F; AC
	91	...	...	...	...	...	...	...	...	94,300	108,000	20.0	N 1575° F; T 1000° F; AC
9% Ni Steel.....	92	...	...	...	...	...	...	...	...	97,200	109,300	21.0	N 1575° F; T 1000° F; AC

<sup>1</sup> N=Normalized; T=Temper; AC=Air Cool.



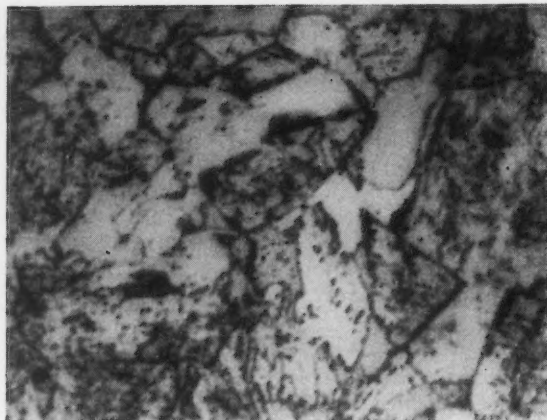


Figure 3—Structure of 9 percent nickel steel showing dark etching constituent at grain boundaries. Nital + picral etch, X 2000.

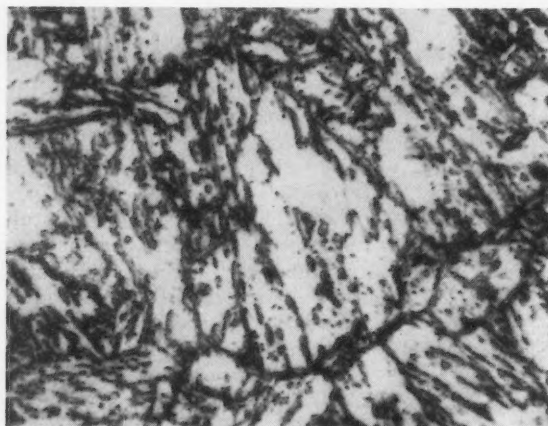


Figure 4—9 percent nickel steel double normalized at 1650 degrees and 1450 degrees F, followed by tempering at 1100 degrees F for two hours. The dark etching constituent is absent. Nital + picral etch, X 2000.

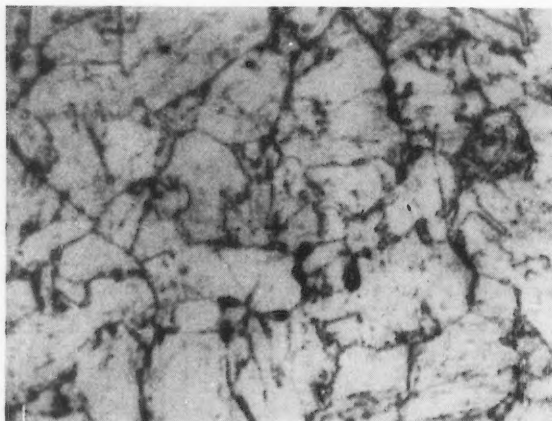


Figure 5—Structure of 9 percent nickel with 0.300 percent titanium; darkened constituent absent. Nital + picral etch, X 2000.

(Test 7, Table I). This treatment is impracticable for the manufacture of tubing and the resulting properties are too low for use where the stresses are high.

The dark etching constituent is also absent in 9 percent nickel steel "stabilized" by the addition of



Figure 6—Crack in martensitic structure of Grade N-80 steel with high manganese content and a hardness of Rockwell C-28 (Test 39). This constituent appears as a network at lower magnification. Nital + picral etch, X 1000.

titanium (Figure 5). This element combines with the carbon of the steel to form titanium carbide, which minimizes or prevents the retention of austenite and thereby reduces or eliminates the subsequent formation of untempered martensite. A prestressed beam specimen of 9 percent nickel steel with 0.30 percent titanium (Test 8) has not failed after 595 days under an applied fiber stress of 96,000 psi. Molybdenum in sufficient amounts may be similarly beneficial, but to a lesser degree. While this appears to be partially confirmed by the resistance of 9 percent chromium-1 percent molybdenum steel to the failure process (Test 17), as compared with the failure of 12 percent chromium steel (Type 410 stainless) in Test 27, the data are not conclusive. Increasing the magnitude of the applied stress and the degree of plastic deformation might sensitize the 9 percent chromium-1 percent molybdenum steel. However, it is definite that stainless Type 410 is a susceptible steel.

The above reasoning applies very well to failures of high nickel steels because they are particularly sluggish in transforming during cooling. It does not explain failure of some other steels that transform much faster so that there is little or no austenite retained for subsequent decomposition. However, this decomposition alone is not necessary for the formation of martensite because this constituent is present in any steel if the rate of cooling, in conjunction with any one of several factors, is fast enough.

All of the steels that failed showed the presence of martensite in some degree of temper, usually in the form of a continuous phase or network. This constituent is shown in Figure 6 which is the structure of the Grade N-80 steel (Test 39) with the higher than normal manganese content and a Rockwell hardness of C28. (Note the fine crack in the martensite.) Tempering this steel at 1000 degrees F, which reduced the hardness to Rockwell C24, did not alter the martensitic network or temper it sufficiently to prevent early failure in the specimen prestressed to the tensile strength and loaded to 80 percent of the yield strength (Test 44).

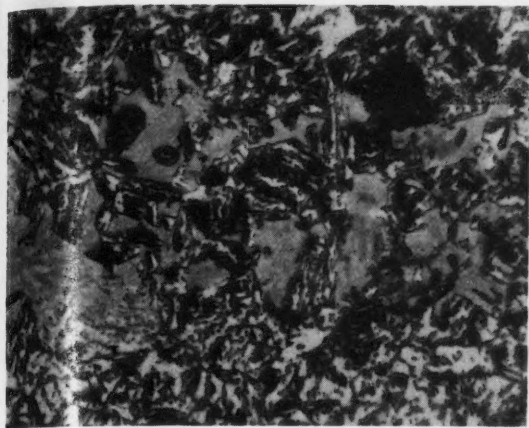


Figure 7—Martensite in Grade N-80 steel (Test 47). This constituent appears as a network at lower magnification. Nital + picral etch, X 1000.

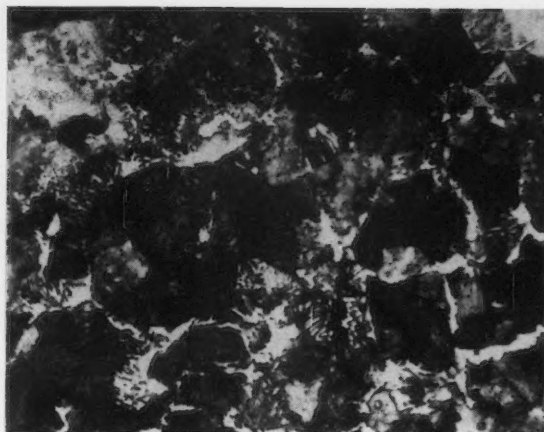


Figure 10—Structure of 5 percent nickel steel showing scattered areas of martensite. Nital + picral etch, X 1000.

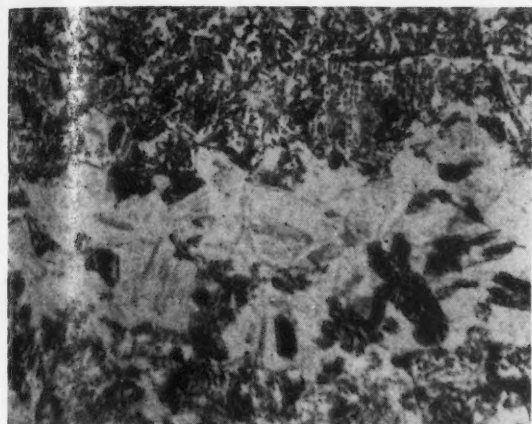


Figure 8—Maximum martensitic condition in substitute Grade N-80 steel (Tests 61 to 64). This constituent was not present generally as a continuous network. Nital + picral etch, X 1000.

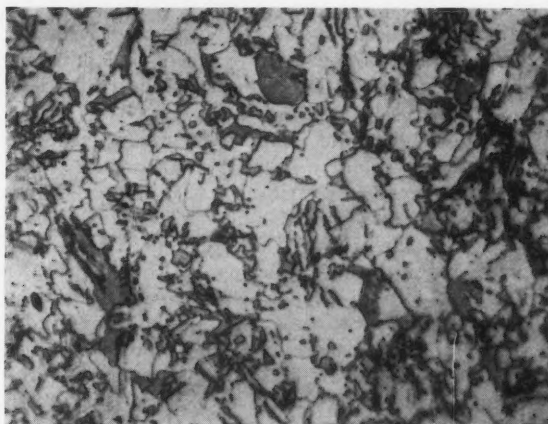


Figure 11—Fully tempered low carbon martensite with spheroidized carbides in 9% chromium-1% molybdenum steel. Nital + picral etch, X 1000.

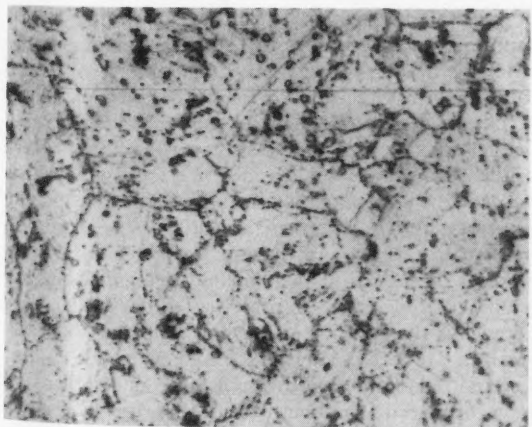


Figure 9—Structure of substitute Grade N-80 steel. No Martensite is present. Nital + picral etch, X 1000.

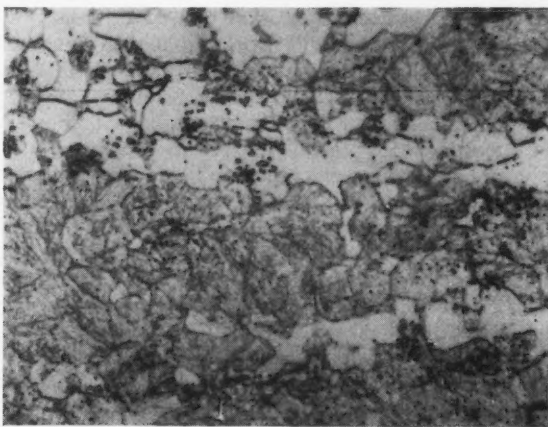


Figure 12—Segregation of martensite in 12% chromium steel (Test 27). Nital + picral etch, X 1000.

Grade N-80 steel in Tests 45 to 48, inclusive, showed a minor amount of this martensitic constituent (Figure 7). Failure occurred after 76 days (Test 47) in the specimen under the most severe conditions of loading and prestressing. The other tests have been in progress for only 79 days and failures may still occur.

The structure of the substitute Grade N-80 steel in Tests 61 to 64 is shown in Figure 8. This represents the maximum martensitic condition present. For the most part there were only minor amounts of the constituent found and these did not appear nearly so much as a network as did the martensite in the

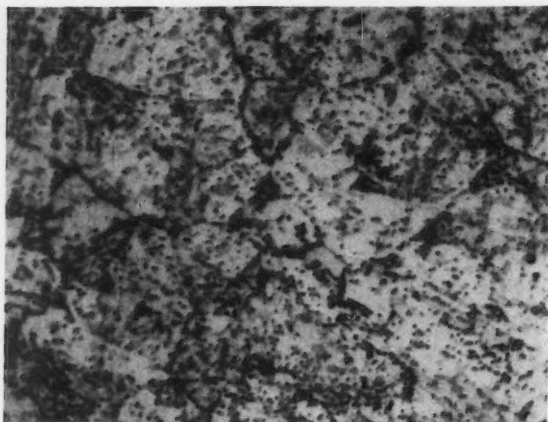


Figure 13—Dark etching constituent in structure of 5% nickel—1% chromium steel. Nital + picral etch, X 2000.

specimens of Tests 39 to 42, inclusive. However, eventual failure of the more severely plastically deformed specimens of the group, represented by Tests 61 to 64, would not be surprising.

The other substitute Grade N-80 steels, except the one noted above, did not exhibit martensite in any degree of temper or distribution. Figure 9 is typical of the structure found. No failures have occurred in these steels. The Grade J-55 steel, heat treated by water quenching and tempering to Rockwell C30 to C31 (Tests 33 and 34), showed the martensite network. Tests on specimens of this steel, tempered at higher temperature to reduce the hardness to Rockwell C22 to C24 (Tests 35 to 38, inclusive) have not been under way long enough to show whether the martensite has been tempered sufficiently to prevent failure.

Not all of the martensitic steels failed. The structure of 5 percent nickel steel (Test 9) in Figure 10 shows some scattered areas of this constituent; but there is no continuous network. This steel has been under test in the prestressed condition for 662 days. The structure of the 9 percent chromium-1 percent molybdenum steel (Test 17) in Figure 11 shows a fully tempered low carbon martensitic structure with spheroidization of the carbides. No failure occurred in this test after 661 days. However, as mentioned before, failure might occur in this steel if the magnitude of the applied stress and the degree of deformation were increased. The structure (Figure 12) of the 12 percent chromium steel, which was shown to be susceptible by failing in 123 days (Test 27), showed segregation of martensite.

All of the alloy steels tested in Table III contain a continuous phase or network of martensite. The structure of 5 percent nickel—1 percent chromium steel (Figure 13), is more or less typical. It should be pointed out again that the 9 percent nickel steels listed in this table exhibited the dark etching constituent shown previously in Figure 3.

#### Discussion

Regardless of any or all possible factors that may be involved, all failures exhibited a network or segre-

gation of martensite. Thus, it appears that a network of martensite of sufficient hardness is necessary to render a steel susceptible to the failure process under the selected conditions of environment. Since the network generally represents only a very small portion of the area, a Rockwell test, involving a relatively large area, would not give a true indication of the hardness of the martensitic structure and, therefore, would not be a reliable means of indicating susceptible steels.

When there is considerable difference in hardness between the network and the remaining structure, plastic deformation increases the susceptibility to failure because the mechanical working naturally results in unequal strains being set up, a positive source of stress-corrosion cracking under the selected conditions. However, when the network is tempered, though not necessarily removed, so that the differential between its properties and the remainder of the structure is reduced sufficiently, plastic deformation consequently does not result in damaging unequal strains and the steel is not susceptible to the failure process. Therefore, plastic deformation, as in cold straightening, would not be expected to have an effect on hot finished steel pipe that does not have retained austenite for decomposition or a martensitic network by virtue of analysis or subsequent heat treatment. The results herein sustain this observation.

Oil country tubing is ordinarily fabricated to meet minimum property specifications. The early work in this investigation indicated that most steels heat treated to produce mechanical properties in or above a critical range represented approximately by Rockwell hardness C24 to C26 may be rendered susceptible to the failure process. This suggested that it might be necessary to place limitations on the maximum properties. In the light of the present work, it is apparent that selection of steel for use under the existing conditions on a basis solely of composition or mechanical properties, either maximum or minimum, is not safe unless the steel is free of a martensitic network or segregation. Obviously, it is impracticable to specify the steel on a basis of the metallographic structure. Therefore, despite the fact that mechanical properties are not a precise control of the susceptibility of the steel to the failure process, it will still be necessary to place maximum mechanical property limits on any given steel that will insure against the presence of damaging martensite in the structure.

Most of the failures of beam specimens that have occurred in this laboratory study have been duplicated in the pilot production field test at Gulf's Walter Marr No. 1 Well, Pincher Creek, Alberta, Canada. Also a few additional failures in other steels have occurred which indicates that the field operating conditions involving pressure, flow, and formation water were more severe than the laboratory conditions selected. It is evident that steels should be tested under the exact service conditions before final selection is made.

#### Summation

The results to date seem to justify the following observations:



1. Ordinary tubing steels (API Grades J-55 and N-80) are at least the equal of low alloy steels under the conditions of loading and environment utilized.
2. The sensitivity of steel to failure in hydrogen sulfide environments results primarily from the presence of martensite in the structure, particularly as a network.
3. Plastic deformation is not essential to cause failure in susceptible steels. However, in such steels the failure process is accelerated as the degree of deformation is increased.
4. Plastic deformation of the degree investigated herein does not affect steels devoid of martensite.
5. Susceptible steels, with or without plastic deformation, will not fail unless the applied stress is above a "critical" amount.
6. Hardness as ordinarily measured is not a precise criterion in determining the susceptibility of steels to the failure process. Other mechanical properties are similarly deficient.
7. The susceptibility of steels to failure may be

retarded or prevented by any change in composition, or by any heat treatment that eliminates or sufficiently tempers a martensitic network.

8. The maximum mechanical properties of a given hot finished steel for service in sulfide environments involving high stresses, should be limited by values that will insure against damaging martensite in the structure of the given steel.

#### Acknowledgment

The authors thank L. W. Vollmer for his many helpful suggestions in carrying on the program and in preparing the manuscript.

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# Cracking of High Strength Steels In Hydrogen Sulfide Solutions\*

By J. P. FRASER and R. S. TRESEDER\*

**T**HE SPONTANEOUS cracking of high strength steels when exposed under tensile loading in aqueous hydrogen sulfide solutions is a corrosion effect of considerable economic importance in the oil industry. This effect has been designated as "sulfide corrosion cracking," such terminology being prompted by the lack of clear evidence that the effect was either stress corrosion cracking or hydrogen embrittlement. Failures of this type have been experienced in the past,<sup>1-6</sup> but little work was done on the problem until recently when it became apparent that the development of certain high pressure sour gas-condensate fields might be seriously hampered if suitable precautions were not taken to prevent such failures. It appeared that sulfide corrosion cracking could be encountered with either casing, tubing, wellhead fittings or flowlines. Owing to the high pressures involved a single failure might have disastrous results.

The lack of adequate field experience on which to base selection of materials for new wells led to an intensive program of laboratory investigation and field testing. This paper presents a summary of the laboratory results thus far obtained. The work is not completed and many questions remain to be answered, but it is believed that certain aspects of the problem have been clarified and an understanding reached as to the mostly likely preventive measures that can be employed.

## Laboratory Test Methods

A laboratory test procedure was devised to allow evaluation to be made of the various environmental, metallurgical and mechanical factors involved in sulfide corrosion cracking. This procedure involved use of a special holder made of Type 316 stainless steel (see Figure 1) wherein a strip specimen, 0.06-in. x 0.25-in. x 3.0-in. was loaded as a simple beam by means of a calibrated loading screw. Beam supports and load contact were made of glass to eliminate galvanic effects between the specimen and holder. In general specimens were tested with a milled surface, degreased just prior to test. The specimen was loaded to a given deflection by means of the calibrated loading screw and then immersed vertically in the test solution (200 ml per specimen) contained in a one-liter rubber-stoppered glass cylinder fitted with a gas dispersion tube. The test solution was initially

## Abstract

Laboratory data are presented relating to the spontaneous cracking and embrittlement of steel alloys under environmental and stress conditions pertinent to sour gas condensate wells. It has been shown that cracking can occur in simple aqueous hydrogen sulfide solutions and is an effect related to stress corrosion cracking. A mechanism involving stress corrosion cracking and hydrogen embrittlement is proposed to explain the cracking phenomenon. Important factors studied included composition and heat treatment of alloy, type and magnitude of stress, Composition of corrosive solution (e.g., acidity, salt content), composition and pressure of gas environment, temperature and time of exposure. Of these the first three were found to be the most important. Remedial measures discussed include use of resistant alloys, heat treatment of susceptible alloys, organic and metallic coatings and inhibition. A simple laboratory procedure is presented for making a preliminary evaluation of the cracking susceptibility of a given alloy.

freed of entrained and dissolved gas by boiling and the solution was subsequently handled under an atmosphere of nitrogen. After immersion of the test holder the solution was saturated with the test gas, usually cylinder hydrogen sulfide. Periodically the solution was re-saturated by bubbling the test gas through for several minutes. There was no other agitation of the solution during the test period. Except where noted, tests were conducted at room temperature (20-25° C). Sometimes several related

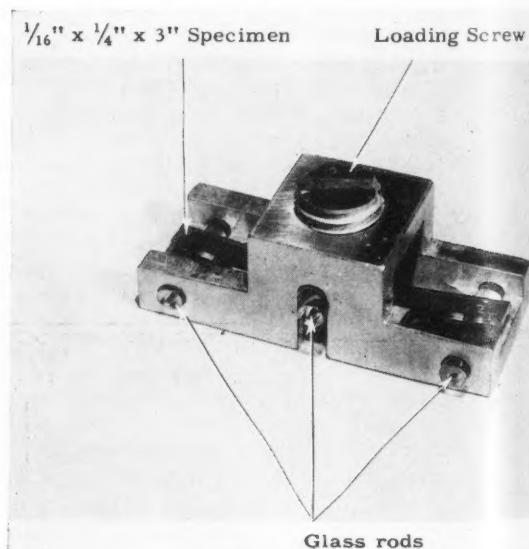


Figure 1—Holder for constant-deformation stressed specimens.

\*A paper presented at the Eighth Annual Conference, National Association of Corrosion Engineers, Galveston, Texas, March 10-14, 1952.

\* Both with Shell Development Company, Emeryville, Cal.

tests were made in the same cylinder, in which case additional solution was used so as to maintain a constant ratio of solution volume to metal surface. Tests were continued for periods up to a maximum of about two months, although in most cases they were terminated after several weeks since with very few exceptions failures occurred within a few days. Time to failure usually was not considered to be pertinent because the test was one in which a particular material either failed or passed. The reproducibility of the test was very good under the more severe conditions, i.e., highly susceptible alloys in a severe environment, but under borderline conditions somewhat erratic results were obtained, particularly with API grade N-80 steels.

The principal advantage of the above test procedure lies in its simplicity, the corrosive system being reduced to its most simple form so that evaluation of the various factors entering into the actual well condition can be made in an independent, systematic manner. An important feature is the use of small specimens which allows samples to be machined directly from regular tubing or wellhead fittings without complicating the test by introducing extraneous mechanical or heat treatments as would be required if larger, specially fabricated samples were employed.

### Environmental Factors

Employing several tubing and wellhead alloys of different mechanical and chemical properties (see Table I) study was made of the influence of environmental factors on susceptibility of these alloys to cracking. Tests were made with a standard fixed-deformation loading equivalent to a nominal outer fiber stress of 120,000 psi. Since this was above the yield strength of most alloys tested, the specimens thereby were subjected to a slight amount of cold work and the actual stresses would be indeterminate. The 120,000 psi nominal stress was chosen as a standard since it would represent a severe, but not unreasonable, condition. Although this stress is in excess of nominal design, such high stresses might be encountered near stress raisers, in local areas under severe operating conditions, or as a result of cold straightening during fabrication. In view of the nature of the failure it was considered that testing under less severe loading conditions might lead to a false sense of security.

It should be noted at this time that the tendency for cracking of a particular alloy is dependent on

several factors other than its chemical composition, as will be established later. Hence, results obtained with a particular stock of an alloy should not be construed as being representative of all materials of that alloy composition.

### Gas Composition

Tests showed that hydrogen sulfide was the gas component responsible for the cracking effect, but that the presence of liquid water was also essential. No cracking was observed on exposure of stressed specimens of a susceptible alloy, e.g., 9 percent nickel steel, to water-saturated hydrogen sulfide gas or to a kerosene solution of hydrogen sulfide. No cracking was observed in tests with aqueous carbon dioxide solutions. Mixtures of carbon dioxide and hydrogen sulfide were found to be no more severe than hydrogen sulfide, i.e., the same alloys cracked in both cases. Presence of oxygen appeared to have negligible effect. Tests employing gas samples from the field gave results comparable to those obtained with cylinder hydrogen sulfide. The above tests were all made at one atmosphere pressure. Tests with an atmosphere of hydrogen sulfide plus carbon dioxide at pressures corresponding to the partial pressures of these gases at the wellhead of a typical sour gas-condensate well gave results similar to those with hydrogen sulfide alone at atmospheric pressure.

### Composition of Aqueous Phase

In those tests made with a simple aqueous solution of hydrogen sulfide, only 9 percent nickel steel failed among the ten alloys tested (Table II). The presence of 0.5 percent added acetic acid resulted in failures of six additional alloy samples (9 percent chromium steel, wrought 12 percent chromium steel, 7 percent chromium steel, 5 percent chromium steel, SAE 4340 steel and N-80). Similar results were obtained with a 0.1 percent acetic acid solution: at 0.01 percent concentration only 9 percent nickel steel, 7 percent chromium steel, 5 percent chromium steel and SAE 4340 steel failed. It is presumptive other water-soluble aliphatic acids would give comparable results.

In a few tests made with sodium chloride solutions at concentrations up to 6 percent, it was found that the addition of chloride had a marked effect on susceptibility to cracking of certain of the chromium steels (Table II).

Analysis of produced water from a typical sour gas-condensate well showed an organic acid concen-

TABLE I—Mechanical Properties and Chemical Compositions of Alloys Tested

ALLOY	CHEMICAL COMPOSITION, PERCENT						INITIAL MECHANICAL PROPERTIES				
	C	Mn	S	P	Si	Other	Yield Str., psi	Ult. Tens. Str., psi	Elong., Percent	Red. of Area, Percent	Rockwell Hardness
9% Ni steel	0.08	0.39	0.023	0.018	0.19	8.82 Ni	106,000	126,000	26.	66.	24C
9% Cr steel	0.09	0.35	0.008	0.018	0.33	8.41 Cr 0.91 Mo	158,000	185,000	20.	58.	38C
12% Cr steel, wrought	0.05	0.45	0.011	0.009	0.55	12.4 Cr	102,000	142,000	24.	63.	28C
7% Cr steel	0.09	0.41	0.006	0.016	0.70	6.65 Cr	126,000	158,000	24.	61.	30C
5% Cr steel	0.08	0.40	0.013	0.015	0.24	4.85 Cr	97,000	140,000	26.	64.	30C
SAE 4340	0.33	0.71	0.010	0.010	0.25	0.75 Cr 1.64 Ni 0.30 Mo	126,000	148,000	20.	56.	27C
N-80 (API grade)	0.40	1.57	0.03	0.017	0.15	0.22 Mo	91,000	118,000	22.	55.	21C
5% Ni steel	0.09	0.43	0.014	0.002	0.27	4.83 Ni	61,000	99,000	36.	69.	52A
1-55 (API grade)	0.40	0.72	0.02	0.003	0.14	.....	54,000	92,000	30.	53.	53A
12% Cr steel, cast	0.08	0.49	0.020	0.023	0.80	12.1 Cr	55,000	84,000	29.	59.	50A



tration of approximately 74 ppm, calculated as propionic acid and a sodium chloride content of about 100 ppm. Laboratory cracking tests with such a water sample saturated with hydrogen sulfide gave results indicating it to be somewhat more severe than distilled water, but less severe than a 0.5 percent acetic acid solution (Table II).

Corrosion rate values obtained in a number of the above cracking test environments are summarized in Table III. There was no general correlation of corrosion rate with cracking tendency. It was noted, however, that for the wrought high chromium steels (7, 9 and 12 percent Cr) the one environment (distilled water saturated with hydrogen sulfide, 25° C) in which cracking did not occur was also the one where no significant corrosion of these alloys was observed. In contrast to this were the results obtained with 0.5 percent acetic acid solutions at varying temperatures, in which increasing corrosion rate occurred with increasing temperature, whereas the cracking decreased with increasing temperature.

### Inhibition

Data obtained from tests with a typical commercial oil field corrosion inhibitor indicate that sulfide corrosion cracking can be inhibited.

### Effect of Liquid Hydrocarbon

The presence of a hydrocarbon phase might be expected to have a protective effect, particularly if the hydrocarbon contained polar compounds, and there are indications from laboratory work that this

is true. Thus in a laboratory test the more severe condition would be to omit the hydrocarbon phase. The influence of this factor becomes a problem in evaluating field test data because the protective nature of the hydrocarbon phase may vary among wells and in addition the manner of exposure may have an effect on the protective action of the hydrocarbon.

### Temperature

Temperature had an appreciable effect on the tendency to crack of borderline materials in those environments tested. The general effect was that of increasing severity with decreasing temperature (Table II). For example, 5 percent nickel steel failed in 0.5 percent acetic acid (hydrogen sulfide saturated) at 0 degrees C but not at 20-25 degrees C.

Because wellhead temperatures during operation generally would be higher than the temperature (20-25 degrees C) employed in the bulk of these laboratory tests, the laboratory tests represent a more severe condition in this respect, but nevertheless one that would be encountered in practice, i.e., during shut in periods.

### Correlation of Laboratory Results With Field Test Results

Field tests have been conducted at the Jumping Pound field in Canada in which stressed specimens were exposed in the flowline of a well. Specimens used were of the same type and same alloy stocks as employed in the laboratory tests. Failures of 9 percent nickel steel, SAE 4340, and 7 percent chromium

**TABLE II—Summary of Cracking Test Results**  
All specimens stressed to 120,000 psi nominal max. fiber stress. Numbers in table refer to number of specimens failed of number tested. Exposure times varied from 5 to 22 days.

ALLOYS Arranged in approx. order of decreasing susceptibility	ENVIRONMENTS (APPROX. ORDER OF DECREASING SEVERITY)									
	1 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S Atm. 0° C	2 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S 25° C	3 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S+CO <sub>2</sub> 25° C	4 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S 80° C	5 Jumping Pound Field Test	6 6% NaCl H <sub>2</sub> S 25° C	7 J.P. Brine H <sub>2</sub> S 25° C	8 Dist. H <sub>2</sub> O H <sub>2</sub> S 25° C	9 0.5% CH <sub>3</sub> COOH CO <sub>2</sub> 25° C	9' Dist. H <sub>2</sub> O CO <sub>2</sub> 25° C
9% Ni steel.....	XXX	26 of 26	XX	XX	XXOO	XXXO	X	21 of 21	OO	OO
9% Cr steel.....	XXX	XX	XX	XX	XX	X	O	OO	...	...
12% Cr steel, wrought.....	XXX	XX	XX	XX	X	X	O	OO	...	...
7% Cr steel.....	XXX	XX	XX	XX	X	X	O	OO	...	...
5% Cr steel.....	XXX	XX	XX	XX	X	X	O	OO	...	...
SAE 4340.....	XXX	XX	XX	XX	XXXO	O	O	OO	...	...
N-80.....	XXXOO	7 of 27	OO	OO	OO	...	...	OO	OO	OO
5% Ni steel.....	XO	OOO	...	...	OOOO	...	...	OO	...	...
J-55.....	OO	OOO	...	...	OO	...	...	OO	...	...
12% Cr steel, cast.....	OO	OOO	...	...	O	...	...	OO	...	...

X = Specimen failed by cracking.

O = No cracking observed.

**TABLE III—Summary of Corrosion Test Results**  
Tests made under conditions comparable with those in Table II, except that solutions were moderately agitated. Unstressed specimens used. Numbers in table refer to average corrosion rate for two specimens. All rates expressed in mils/yr.

ALLOYS Arranged in approx. order of decreasing susceptibility to cracking	ENVIRONMENTS (ARRANGED IN APPROX. ORDER OF DECREASING CRACKING SEVERITY)							
	1 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S Atm. 0° C 1 day	2 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S 25° C 1 day	3 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S+CO <sub>2</sub> 25° C 1 day	4 0.5% CH <sub>3</sub> COOH H <sub>2</sub> S 80° C 1 day	5 Jumping Pound Field Tests 2 weeks	6 6% NaCl H <sub>2</sub> S 25° C 1 day	7 J.P. Brine H <sub>2</sub> S 25° C 2 weeks	8 Dist. H <sub>2</sub> O H <sub>2</sub> S 25° C 1 day
9% Ni steel.....	8.	12.	17.	172.	...	21.	1.	17.
9% Cr steel.....	12.	68.	...	75.	...	9.	0.4	0.
12% Cr steel, wrought.....	22.	65.	70.	52.	...	3.	0.03	0.
7% Cr steel.....	17.	62.	68.	181.	1.	14.	0.2	3.
5% Cr steel.....	16.	45.	70.	211.	...	23.	0.3	11.
SAE 4340.....	17.	47.	...	335.	...	18.	...	19.
N-80.....	15.	89.	62.	158.	7.	5.	...	28.
5% Ni steel.....	11.	22.	...	212.	2.	22.	...	28.
J-55.....	16.	59.	...	143.	5.	...	...	25.
12% Cr steel, cast.....	18.	37.	...	259.	0.4	5.	...	2.

Note: In each column the horizontal line separates those alloys which failed in standard cracking tests in the particular environment from those which did not fail. Alloys which did not fail are below the line.

steel were observed, but J-55, N-80, 5 percent nickel steel and cast 12 percent chromium steel showed no tendency for sulfide corrosion cracking. These test results are in fair agreement with laboratory tests made with a produced water sample from this same well (Table II). The slight differences noted could be ascribed to differences in gas composition, pressure and temperature. Field conditions appeared to be of intermediate severity compared to the various laboratory test conditions employed. For example, conditions of the Jumping Pound test were apparently less severe than laboratory tests made with 0.5 percent acetic acid solution, but more severe than such tests with distilled water. Thus it is seen that field test conditions can be approximated reasonably well in the laboratory with relatively simple test procedures.

The general corrosion rate values obtained in these field tests were of the same order of magnitude, i.e., less than 10 mils per year, as those obtained in the comparable laboratory tests with the produced water sample.

### Metallurgical and Mechanical Factors

#### Evaluation of Common Tubular and Wellhead Alloys

Laboratory tests with a variety of possible tubing and wellhead steel alloys indicate that alloy composition, except as it affects hardenability, is a relatively minor variable in determining the susceptibility of an alloy to sulfide corrosion cracking (see Table IV). The common tubing materials, API grade N-80 and J-55 steels, were found to be more resistant to cracking than some of the more highly alloyed and higher strength, materials such as 9 percent nickel steel and 9 percent chromium steel. Cracking was observed under the more severe test conditions with an N-80 steel whereas a J-55 steel did not fail in any test. A cast 12 percent chromium steel with moderately low mechanical properties was also found to be resistant to cracking, although a sample of the wrought form of this alloy having higher mechanical properties was one of the more susceptible materials tested. The data indicate that there is a rough correlation between hardness and susceptibility to cracking, although it is believed that caution should be exercised in using hardness alone as a criterion for susceptibility of an alloy.

A study of more highly alloyed materials for possible use in wellhead fittings indicated that the austenitic stainless steels, Monel, K Monel, and Stellites No. 1 and No. 6 were resistant to sulfide corrosion cracking. All of the stainless alloys were not resistant, however; the precipitation-hardening as well as the martensitic grades of stainless steels were found to be quite susceptible to this type of failure, especially if chloride ion were present (see Table V).

#### Heat Treatment

Mill practice in the production of both oil well tubular goods and wellhead fittings varies considerably, as does the chemical composition of these ma-

TABLE IV—Correlation of Mechanical Properties and Relative Susceptibility of Alloys to Sulfide Corrosion Cracking

ALLOY (Listed in approx. order of decreasing susceptibility to cracking.)	Rockwell Hardness	Yield Strength, psi	Ult. Tensile Strength, psi
9% Ni steel.....	24C	106,000	126,000
9% Cr steel.....	38C	158,000	185,000
12% Cr steel, wrought.....	28C	102,000	142,000
7% Cr steel.....	30C	126,000	158,000
5% Cr steel.....	30C	97,000	140,000
SAE 4340.....	27C	126,000	148,000
N-80.....	21C	91,000	118,000
5% Ni steel.....	52A	61,000	99,000
J-55 <sup>1</sup> .....	53A	54,000	92,000
12% Cr steel, cast <sup>1</sup> .....	50A	55,000	84,000

<sup>1</sup> No failures in any tests to date.

TABLE V—Susceptibility of Various Alloys to Sulfide Corrosion Cracking

0.5% acetic acid solutions. If no failures occurred within one week, 6.0% w NaCl was added. Total exposure time, 3 weeks. H<sub>2</sub>S atmosphere, 20-25° C. Nominal max. fiber stress = 120,000 psi.

ALLOY	Condition	Rockwell Hardness	Test Results X = Failure by Cracking O = No Cracks Observed
Type 302 stainless.....	As received	50A	OO
Type 302 stainless.....	Cold rolled	41C	OO
Type 302 stainless.....	Sensitized at 1200 °F.	50A	O
Type 316 stainless.....	As received	48A	OO
Type 410 stainless.....	As received	53A	XX
Type 410 stainless.....	Quenched from 1800 °F.	40C	XX
Type 430 stainless.....	As received	47A	OO
17-4 pH stainless <sup>2</sup> .....	As received	31C	XX <sup>1</sup>
17-4 pH stainless <sup>2</sup> .....	Precipitation-hardened	40C	XX <sup>1</sup>
17-7 pH stainless <sup>3</sup> .....	As received	55A	OO
17-7 pH stainless <sup>3</sup> .....	Precipitation-hardened	32C	XX <sup>1</sup>
Monel.....	As received	53A	OO
K monel.....	Precipitation-hardened	32C	OO
Stellite No. 1.....	As received	60C	O
Stellite No. 6.....	As received	45C	OO

<sup>1</sup> Failures occurred after addition of salt.

<sup>2</sup> 17 Cr, 4 Ni, 4 Cu.

<sup>3</sup> 17 Cr, 7 Ni, 1 Al.

TABLE VI—Effect of Heat Treatment on Resistance of Certain Alloys to Sulfide Corrosion Cracking

0.5% w acetic acid solution used for tests with N-80 and J-55; distilled H<sub>2</sub>O used for tests with 9% Ni steel. H<sub>2</sub>S atmosphere, 20-25° C. Nominal max. fiber stress = 120,000 psi.

Specimens machined from sections of tubing after hardening by heating ½ hr. at temperature indicated, quenching in oil, and tempering for 1 hr. at temperature indicated. Tensile tests made on 0.632 in. long x 1/40 sq. in. round tensile bars cut from tubing wall.

ALLOY	Hardening Temp., °F.	Tempering Temp., °F.	Rockwell Hardness	Yield Str., psi	Ult. Tens. Str., psi	Elong., %	Red'n of Area, %	TEST RESULTS <sup>1</sup>	
								Without Stress Raisers	With Stress Raisers <sup>2</sup>
N-80.....	As received		21C	91,000	118,000	22	55	7 of 27	000
N-80.....	1600	1150	23C	112,000	121,000	26	65	0	000
N-80.....	1600	1100	28C	124,000	140,000	21	59	0	X
J-55.....	As received		53A	54,000	92,000	30	53	00	00
J-55.....	1600	700	27C	99,000	132,000	22	67	0	0
J-55.....	1600 WQ <sup>3</sup>	850	31C	141,000	153,000	14	44	X	X
9% Ni steel.....	As received		24C	106,000	126,000	26	66	21 of 21	XX
9% Ni steel.....	Tempered 64 hr. at 1100 °F.		20C	74,000	126,000	27	71	0	X

<sup>1</sup> X = specimen failed by cracking.

0 = No cracking observed.

<sup>2</sup> Stress raisers consisted of two No. 60 holes drilled on center line near middle of specimen.

<sup>3</sup> Water quenched.



Figure 2—Holder for constant-load stressed specimens.

TABLE VII—Effect of Stress on Sulfide Corrosion Cracking of 9% Nickel Steel

Distilled water,  $H_2S$  atmosphere, 20-25° C., Test duration, 12-22 days.

Nominal Max. Fiber Stress, psi	TEST RESULTS X=Failure by Cracking O=No Observed Cracking
48,000	O O O O
60,000	X O O
72,000	X X X X X O
120,000	21 of 21

materials. Typical production practices for three common materials are as follows:

1. J-55—Hot rolled. Forging temp. = ca. 2180 degrees F.
2. N-80—a) Normalized from 1600 degrees F or  
b) Cold drawn and stress relief annealed.
3. Cast 12 percent Cr steel—Sand cast and cooled in mold. May be tempered at ca. 1200 degrees to 1300 degrees F.

It is obvious that the mechanical properties and microstructures of these alloys will vary over a considerable range, depending on the particular metallurgical history involved; and the question is immediately raised as to the effects which these factors will exert on the susceptibility of the alloys to sulfide corrosion cracking.

Because high physical properties are associated with susceptibility to cracking, it would seem that an annealing type of heat treatment should lead to an increase in resistance of an alloy to this type of failure. The data for the 9 percent nickel steel in Table VI indicate that this is so; but an annealing heat treatment also leads to a reduction in yield and ultimate strengths, which is undesirable from a de-

sign standpoint. For the low alloy steels, it has been found that the susceptibility to cracking is closely related to the microstructure; a quenched-and-tempered microstructure has been found to have markedly better resistance than a normalized microstructure at the same level of mechanical strength and ductility (Table VI).

### Stress

The probability of failure by sulfide corrosion cracking in general is related to the magnitude of the applied tensile stresses, as is shown by Table VII. Using a particular stock of 9 percent nickel steel exposed to an aqueous hydrogen sulfide solution no failures were observed when the specimen was stressed to 40,000 psi, but failures did occur at stress values of 60,000 psi and higher, the percentage of failures increasing with the stress. Stress raisers and cold bending, since they increase the magnitude of the tensile stresses at the surface of the metal, also increase the probability of failure. Increasing applied stresses will not insure failure if the alloy has an extremely low susceptibility to failure, such as the J-55 steel.

The majority of the data here reported has been obtained using constant-deflection loading. In tests with specimens stressed by means of a constant applied load rather than using a fixed deformation, it was found that failures occurred at appreciably lower stresses. The apparatus shown in Figure 2 was used for these tests, the load being transmitted to the specimen by means of a Type 316 stainless steel spring.

### Protective Coatings

Plastic and metallic coatings were found to be protective to steel only if there were no holidays in the coating at areas of high tensile stress. For example, specimens completely coated with a vinyl coating or coated only in the area of high tensile stress were protected by the coating, whereas even a 1/32-inch wide gap in the coating on the tension side of a specimen near the point of maximum stress permitted failure by sulfide corrosion cracking (see Table VIII).

### Mechanism of Sulfide Corrosion Cracking

Three mechanisms for failure by sulfide corrosion cracking are believed to be possible: 1) internal hydrogen stressing, 2) stress corrosion cracking, and 3) a combination of these two. The mechanism of

TABLE VIII—Sulfide Corrosion Cracking Effects with Partially Coated Specimens

Distilled water,  $H_2S$  atmosphere, 20-25° C. Specimens of 9% Ni steel coated with an air-drying vinyl enamel.

DETAILS OF COATING	Nominal Max. Fiber Stress, psi	Time to Observed Cracking, Days <sup>1</sup>
Completely coated	120,000	> 27
Tension side and edges coated, compression side bare	120,000	> 6
1/32-in. wide holiday in center of tension side, remainder of specimen coated	120,000	1
1/2-in. wide strip coated in center of tension side	100,000	4 <sup>2</sup>
1/2-in. wide strip coated in center of tension side	82,000	> 12

<sup>1</sup> > Indicates no cracking in test period.

<sup>2</sup> Cracked just outside of coated area. Estimated stress at point of failure = 80,000 psi.



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cracking due to internal hydrogen stressing involves, first, occlusion of a part of the hydrogen generated by the corrosion reaction as atomic hydrogen or protons and, secondly, precipitation within the metal of molecular hydrogen at lattice dislocations and inclusions. Very considerable gas pressures and resultant stresses can be developed at such sites.<sup>7</sup> These stresses when added to existing internal and applied stresses conceivably could cause failure by sudden cracking. A stress corrosion mechanism postulates that the corrosive attack will proceed along certain more or less continuous paths and at right angles to the applied tensile stresses until failure occurs. At least one function of the tensile stresses is to keep the already-formed crack open, although the stresses will also cause the root of the crack to be anodic to the main surface area of the metal. A combination theory would postulate initiation of the crack by stress corrosion followed by acceleration of the rate of propagation of the crack due to hydrogen released in the vicinity of the root of the crack.

The experimental observation that lowered temperatures and increased acidity appear to favor both sulfide corrosion cracking and hydrogen embrittlement lend some support to the internal hydrogen stressing mechanism for failure. The principal characteristic of sulfide corrosion cracking failures that distinguishes them from failures by hydrogen embrittlement, however, is that materials having appreciable residual ductility may crack on exposure to the corrosive fluids under applied stresses near or below the yield point. For example, a steel having an original reduction of area in the tension test of 66 percent still had enough ductility after exposure to aqueous hydrogen sulfide solutions for one day to give a reduction of area of 17 percent. This same steel has been made to fail fairly consistently in sulfide corrosion cracking tests when loaded to only 70 percent of the yield stress. In addition, there does not appear to be any correlation between the susceptibility of a given material to embrittlement and to cracking, which argues against the hydrogen embrittlement mechanism for sulfide corrosion cracking (see Table IX).



Figure 3—Typical sulfide corrosion cracking of 9 percent nickel steel. Specimen exposed one day in distilled water, H<sub>2</sub>S atmosphere, room temperature.

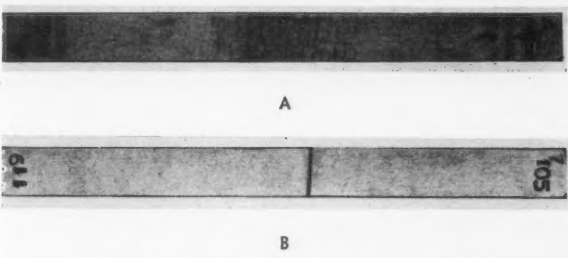


Figure 4—Comparison of stress corrosion cracking with sulfide corrosion cracking—macroscopic aspects. A. Stress corrosion cracking of N-80 steel. Two days' exposure to 0.7 percent HCN, air atm., 20-25 degrees C. B. Sulfide corrosion cracking of N-80 steel. One-day exposure to 0.5 percent acetic acid, H<sub>2</sub>S atm., 20-25 degrees C.

Stronger evidence against the internal hydrogen stressing mechanism is the data obtained with beam-type specimens partially covered with a vinyl coating (Table VIII). In one series of tests stressed specimens of a susceptible alloy coated only on a narrow section opposite the point of loading were exposed in a typical environment. At high loads the specimen cracked just outside the coated area; at lower loads, which were still high enough to produce failures in uncoated specimens, no failure occurred. From previous experience it was known that such a specimen would be as embrittled as one without any coating; if embrittlement were the sole factor involved, failure would have been expected. In the reverse test wherein the stressed specimen was completely coated except for a narrow holiday opposite the point of loading, failure occurred as quickly as with an uncoated specimen. This result also argues against the internal hydrogen stressing mechanism and favors the stress corrosion mechanism.

Sulfide corrosion cracking is apparently related to stress corrosion cracking in that similar conditions are required for producing the effect, i.e., applied tensile stress and exposure to a corrosive environment and also in the nature of the failure, i.e., rapid crack propagation without pronounced metal loss. Certain differences exist; for example, in sulfide corrosion cracking tests with simple beam loaded specimens there is generally only one macroscopic crack, whereas in stress corrosion cracking it is more common to find numerous cracks (see Figures 3, 4 and 5).

At least one specimen from each of the tubing alloys tested in the cracking tests has been examined metallographically. These examinations revealed: 1) the cracking was predominately transgranular, and 2) microscopic cracks were usually present whether or not there was macroscopic cracking. These microscopic cracks started at the bottom of small pits on the tension side of the specimen; typical examples are shown in Figures 6, 7 and 8. In addition, it was found that specimens which exhibited the typical

TABLE IX—Susceptibility of Various Steels to Hydrogen Embrittlement Under Conditions Employed in Cracking Tests

Tensile tests made on 0.632-inch gage length x 1/40 sq. in. round tensile bars in Hounsfield tensometer within ten minutes after removal from the test solution. H<sub>2</sub>S atmosphere. Exposure time, 22 1/2 hours.

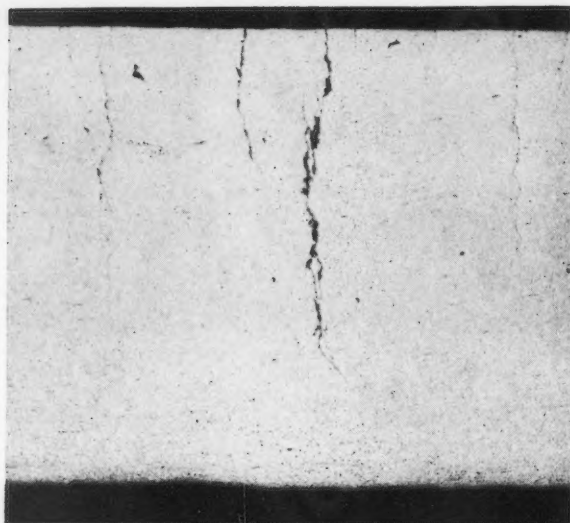
Note: In each column the horizontal line separates those alloys which failed in standard cracking tests in the particular environment from those which did not fail. Alloys which did not fail are below the line.

ALLOYS Arranged in ap- prox. order of decreasing sus- ceptibility to cracking.	Red'n of Area, % as Rec'd	LOSS IN DUCTILITY, PERCENT <sup>1</sup> AFTER EXPOSURE TO:			
		0.5% CH <sub>3</sub> COOH 0° C.	0.5% CH <sub>3</sub> COOH 25° C.	0.5% CH <sub>3</sub> COOH 80° C.	Dist. H <sub>2</sub> O 25° C.
		77	100	84	74
9% Ni steel	66	77	77	66	74
9% Cr steel	58	..	100	84	3
12% Cr steel	..	..	..	..	..
wrought	63	98	100	76	14
2% Cr steel	61	..	98	56	32
9% Cr steel	64	..	96	52	49
SAE 4340	56	89	92	20	65
N-80	55	75	57	29	49
7% Ni steel	69	..	80	55	68
1-55	53	..	77	38	73
12% Cr steel, cast	59	78	75	..	0

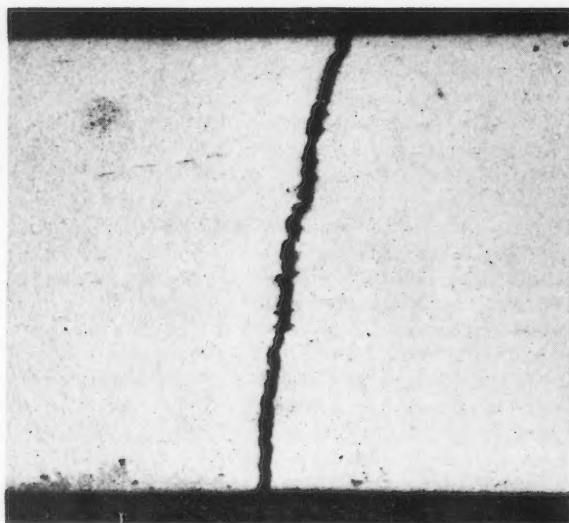
<sup>1</sup> Loss of ductility (%) = (Original reduction of area) — (Final reduction of area) x 100

(Original reduction of area)

Figures given are average of duplicate tests.



A



B

Figure 5—Comparison of stress corrosion cracking with sulfide corrosion cracking—microscopic aspects. Cross sections of specimens shown in Figure 4. X 50.

single macrocrack characteristic of sulfide corrosion cracking usually had microscopic cracks and pitting on the tension side of the specimen (see Figure 9). It is of interest to note that the J-55 steel showed markedly fewer cracks than any of the other alloys. This may be ascribed to the softness of this alloy and its probable lack of notch sensitivity.

Results of metallographic examinations strongly favor stress corrosion as the primary mechanism for failure because the observed pitting and small cracks starting from the bottom of these pits are fairly typical of stress corrosion cracking.

In view of the extreme rapidity of some of the failures in the field and in the laboratory, as well as the presence of but a single macroscopic crack on all of the specimens which failed, it is believed that stress corrosion is not the sole mechanism involved

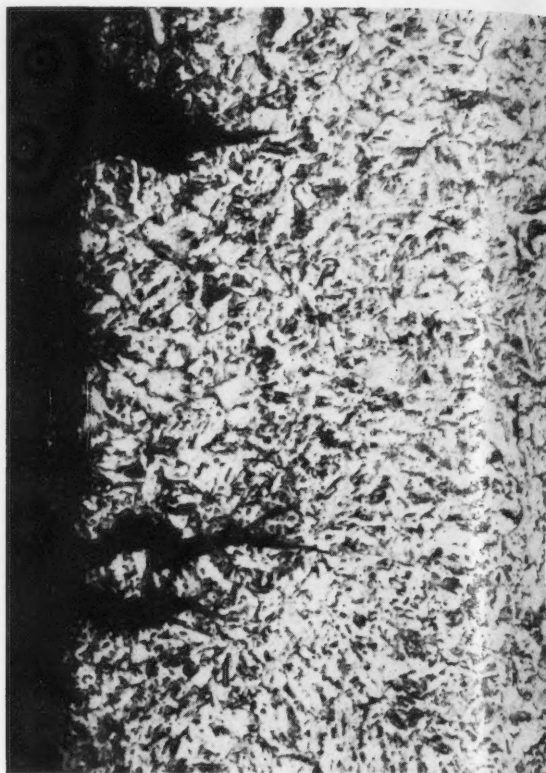


Figure 6—Typical pitting associated with sulfide corrosion cracking of N-80 steel. Tension side of specimen. Six days' exposure to  $H_2S$  atmosphere, 0.5 percent w. acetic acid solution, 20-25 degrees C. Etchant 1.5 percent Nital, X 500.

in sulfide corrosion cracking. The actual mechanism is believed to involve three steps:

- 1) initiation of a very small crack through stress corrosion at some point of high tensile stress (these are usually found at the base of pits in areas under tension),
- 2) the metal at the root of the crack becomes more highly stressed due to notch effects, resulting in localized lattice dislocations which permit occluded atomic hydrogen to precipitate as molecular hydrogen, and
- 3) the additional stress produced by the precipitated molecular hydrogen accelerates the rate of propagation of the crack through the metal until failure occurs.

This mechanism of failure does not preclude the formation of many microscopic cracks on the tension side of a specimen, but before any have gone very far, one will tend to propagate more rapidly than the rest; thus the probability is very low of ever finding more than one macroscopic crack in any given specimen.

### Summary

Laboratory investigation of the phenomenon of sulfide corrosion cracking under conditions pertinent to high pressure sour gas wells shows that the high-strength low alloy steels are all susceptible to this type of failure, the susceptibility of a given alloy being a function of its mechanical properties, micro-

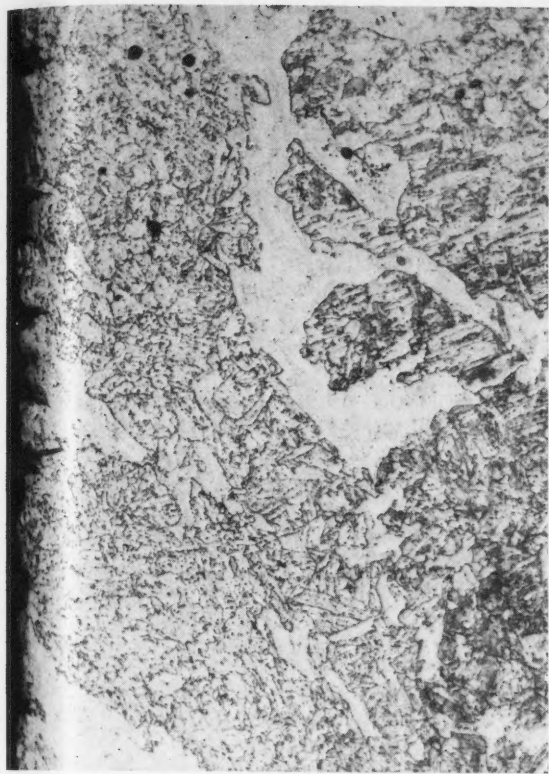


Figure 7—Typical pitting associated with sulfide corrosion cracking of cast 12 percent chromium steel. Tension side of specimen. Six days' exposure to  $H_2S$  atmosphere, 0.5 percent w. acetic acid solution, 20-25 degrees C. Etchant Marble's reagent, X 250.

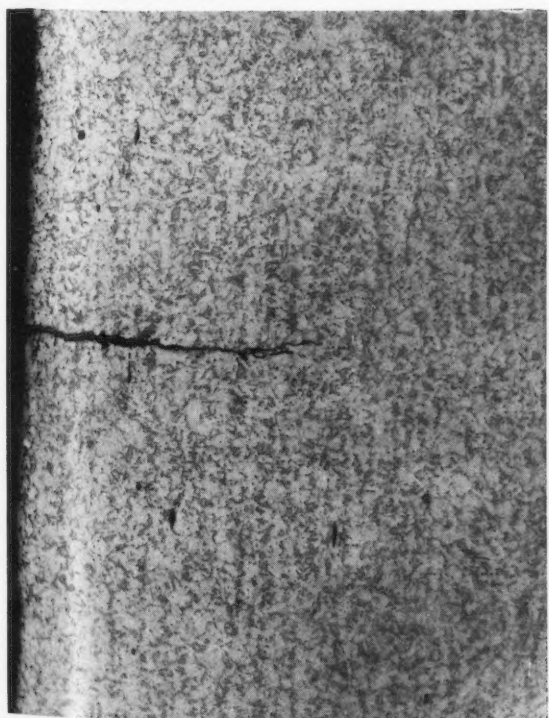


Figure 8—Sulfide corrosion crack in 5 percent nickel steel. Tension side of specimen. Six days' exposure to  $H_2S$  atmosphere, 0.5 percent w. acetic acid solution, 20-25 degrees C. Etchant, 1.5 percent Nital, X 250.

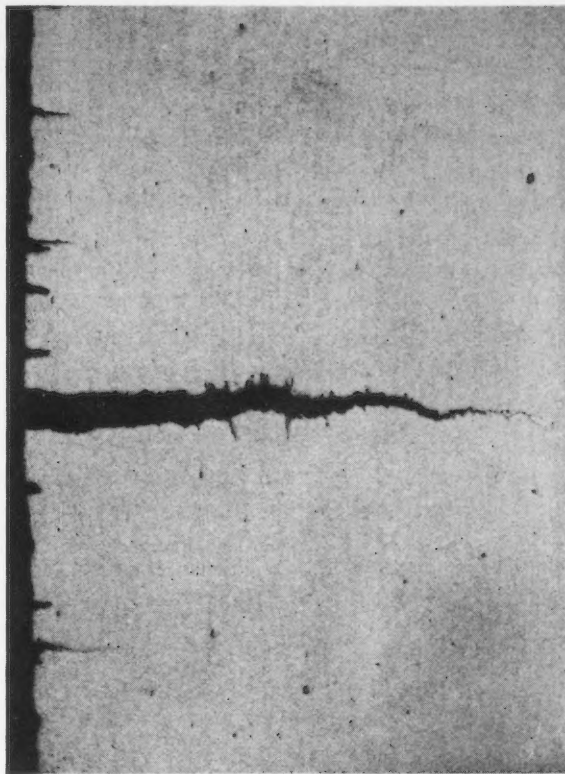


Figure 9—Typical sulfide corrosion cracking of 9 percent nickel steel. Specimen exposed five days in 0.5 percent w. acetic acid solution,  $H_2S$  atmosphere, 80 degrees C. Etchant, 4 percent Nital, X 50.

structural characteristics and alloy content. In general, high mechanical properties (e.g., hardness) are associated with high susceptibility to sulfide corrosion cracking, although at the same hardness level a quenched-and-tempered microstructure appears to be markedly less susceptible than a normalized microstructure.

Increased organic acidity favors sulfide corrosion cracking. Chloride ion increases the severity of the environment towards the chromium steels. There is a greater probability of failure at low than at high temperatures.

Inhibition of sulfide corrosion cracking appears to be possible.

Increased stress, whether from applied load, stress raisers, or cold bending, increases the probability of failure of any given alloy by sulfide corrosion cracking. An annealing type of heat treatment, which lowers the internal stresses in a metal, will thus lower its susceptibility to failure. Laboratory data indicate that constant-load testing is more severe than constant-deformation testing.

Of the ordinary tubing and wellhead alloys, samples of a J-55 and a soft cast 12 percent chromium steel were found to be completely resistant to cracking. A sample of an N-80 steel was found to be borderline in susceptibility to this type of failure. The austenitic stainless steels, Monel, K Monel and



Stellites No. 1 and No. 6 were found to be resistant to sulfide corrosion cracking, but the precipitation-hardening and martensitic grades of stainless steel were susceptible.

Protective coatings would not provide reliable protection against sulfide corrosion cracking because holidays may become sites for initiation of cracks.

Based on the laboratory test data plus metallographic examination, it is proposed that the mechanism of sulfide corrosion cracking involves

- 1) pitting of the surface and initiation of small cracks on the tension side of the specimen,
- 2) local precipitation of occluded hydrogen at or near the roots of the cracks, and
- 3) acceleration of the rate of propagation of the cracks due to the added stresses generated by the precipitated hydrogen until one crack has propagated completely through the specimen.

### Acknowledgment

The authors acknowledge the cooperation of their colleagues in Shell Oil Company and Shell Development Company, especially the guidance of A. Wachter and the contributions of T. M. Swanson, E. W. Wallace, and T. S. Zajac.

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# NACE TECHNICAL PRACTICES COMMITTEE REPORTS

## Field Experience With Cracking of High Strength Steels In Sour Gas and Oil Wells\*

A Report of Technical Practices Committee I-G

**SULFIDE CORROSION** cracking became a corrosion problem of serious concern in the oil and gas production industry about two years ago after the failure of alloy tubing in a well in one of the new sour gas-condensate fields in Canada. Seriousness of the problem was based on the magnitude of the hazard that was implied by this failure; a single failure of the casing or tubing in these high pressure wells could lead to loss of the well and extensive damage to the formation. The number of wells that might be involved, however, would be relatively small since at present there are only few sour gas-condensate fields.

Comparison of experience among various operating companies showed that several similar failures had occurred in other sour gas-condensate wells, although the evidence for corrosion failure as contrasted to mechanical failure was not as pronounced. The striking similarity among all the failures was that they occurred with high strength steel alloys. In this regard there was a similarity to failures observed in the past with steel springs, Bourdon tubes, and hardened valve parts in sour crude or sour gas service in refineries. The latter types of failures had been thought to be a form of a hydrogen embrittlement. The remedial measures employed for these minor equipment failures involved replacement with a resistant alloy such as K-Monel or Type 316 stainless steel. For obvious economic reasons such measures would not be applicable to the problem involving casing and tubing.

The future development of sour-gas condensate fields in Canada and the United States was threatened by the implications of this initial corrosion experience. High strength alloys with mechanical properties equivalent to N-80 or higher were required for tubing and casing because of the well depths and pressures and the meager experience available at that time indicated that it was these high strength alloys that were most susceptible to sulfide corrosion cracking. In an effort to resolve this dilemma the operating companies involved undertook extensive laboratory investigations of this problem coordinated with field testing. In addition the operating companies and suppliers have joined in a cooperative study through the medium of the National Association of Corrosion Engineers. In late 1950 Technical Practice Committee 1G was organized for this purpose. This paper is a report of the initial activities of this committee.



First Pincher Creek failure occurred at 14 inches from upset at depth of 26 feet.

Second Pincher Creek failure occurred at about center section of joint at a depth of 1201 feet.

Figure 1—Failure of 9 percent Ni tubing. (Failure No. 1.)

### Abstract

This is a report by Committee TP-1G on accumulated field experience of several companies with corrosion cracking of oil well tubular goods and wellhead fittings in sour gas and oil wells. This cracking effect has been associated principally with use of high-strength steels in sour gas-condensate wells. Although relatively few failures have been experienced, the problem is a serious one in view of the high pressures involved and the rapid, unpredictable nature of the corrosive attack. Descriptions of failed tubing, casing, and wellhead fittings are given together with data obtained from stressed specimens of various alloys placed in flowlines of wells in several fields. These data show that sulfide corrosion cracking is associated with alloys of high strength, i.e., high hardness, and that the effects may vary among individual wells owing to differing producing conditions and fluid compositions. Possible remedial measures are discussed.

The failures being investigated by this committee could not be characterized easily by current terminology. The failures were not considered to be of the corrosion fatigue type and there was some argument as to whether they were stress corrosion failures or hydrogen embrittlement failures. It was decided, therefore, to give this type of failure the

\*A report presented at the Eighth Annual Conference and Exhibition, National Association of Corrosion Engineers, Galveston, Texas, March 10-14, 1952 by R. S. Treseder, Chairman, NACE Technical Practices Committee I-G on Sulfide Stress Corrosion.

noncommittal designation, "sulfide corrosion cracking." This term implies that the failures are of a spontaneous nature which occurs with certain metal equipment items when exposed under stress (internal or external, or both) to environments containing hydrogen sulfide. When more information is obtained regarding the character of these failures, better terminology should be possible.

### Field Experience

A questionnaire was circulated to those operating companies known to have experienced sulfide corrosion cracking of well equipment. A summarized account of this experience is given below, the information being presented in the form of individual case histories.

#### No. 1

Failed item—9 percent nickel steel tubing  
Well location—Pincher Creek Field, Alberta, Canada.  
Depth—9,200 ft.  
Wellhead pressure—shut in—3200 psig.  
Bottom hole temperature—188 degrees F.  
Wellhead temperature—flowing—96 degrees F.  
Gas analysis—10 percent  $H_2S$ , 6 percent  $CO_2$   
Water analysis—17,000 ppm  $Cl^-$ , pH 7.1  
Water production—300 bbl/day  
Material in annulus—Diesel oil

**Description of failure:** Well had been flowing on a 6-day production test with warm Diesel oil circulating in annulus. Well was shut in and water at ca 50 degrees F was being injected into the annulus to displace the Diesel oil prior to killing the well when failure of the 9 percent Ni steel (2½-inch) occurred at a depth of 26 ft. A subsequent failure occurred at 1200 ft. on pulling the tubing. Maximum applied stress on tubing calculated to be in range 57,000-89,000 psi. Figure 1 presents photographs of the failed tubing. Metallographic examination showed failure to originate on internal surface; cracks were predominantly intercrystalline.

**Remedial measures:** Nickel alloy tubing replaced with phenolic resin coated N-80 tubing. Well shut in except for reduced intermittent flow for test purposes. New tubing has given satisfactory performance under these limited conditions for 2½ years.

#### No. 2

Failed item—9 percent nickel steel tubing  
Well location—McKamie-Patton Field, Arkansas  
Depth—9200 ft.  
Wellhead pressure—shut in—2500 psig.  
Bottom hole temperature—230 degrees F.  
Wellhead temperature—flowing—145 degrees F.  
Gas analysis—7.5 percent  $H_2S$ , 4 percent  $CO_2$   
Water analysis—Substantially no salt content  
Water production—1 bbl/day  
Material in annulus—Bunker fuel.

**Description of Failure:** Tubing pup joint, 2 feet x 2½ inches, of 9 percent nickel steel installed at top of regular tubing string, following which well was swabbed in and flowed for 2 hours; well shut in 30 min., flowed again for 8 hours, and then shut in for 5 hours when failure of tubing pup joint occurred. Metallographic examination showed intergranular cracking, principally on exterior portion.

**Remedial measures:** N-80 and J-55 tubing had never failed in this manner in these wells. Above pup joint replaced with 5 percent nickel steel full length double pup joint, which has given satisfactory service.

#### No. 3

Failed item—9 percent nickel steel tubing  
(Well location and conditions as in No. 2 above)

**Description of failure:** Double pin sub, 1 foot x 2½ inches, machined from 9 percent nickel steel bar stock, installed in tubing string, well swabbed in and flowed for 2 hours, and then shut in for 30 minutes, flowed again for 3 hours when failure occurred. As in No. 2 above metallographic examination showed intergranular cracking, principally on exterior portion.

**Remedial measures:** (As given in No. 2 above.)

#### No. 4

Failed item—N-80 casing  
Well location—Jumping Pound Field, Alberta, Canada  
Depth—10,500 ft.  
Wellhead pressure—shut in—3000 psig.  
Bottom hole temperature—175 degrees F.  
Wellhead temperature—flowing—79 degrees F.  
Gas analysis—3.5 percent  $H_2S$ , 6.5 percent  $CO_2$   
Water analysis—42,000 ppm  $Cl^-$   
Water production—3 bbl/day  
Material in annulus—None. Completed open ended

**Description of failure:** Very little is known regarding this failure which occurred while well was flowing during initial operation. No positive assurance that failure was not purely mechanical although observers at the time characterized the break as brittle. Well subsequently shut in.

**Remedial measures:** Annulus to be packed off to protect casing. N-80 had not failed previously in similar service.

#### No. 5

Failed item—Wire line.  
(Well location as in No. 4 and well conditions substantially the same)

**Description of failure:** Steel wire line failed on at least three occasions. Very little known about nature of failure.

**Remedial measures:** None.

#### No. 6

Failed item—Hardened steel Bourdon tube  
Well location—Columbia City, Arkansas  
Depth—9000 ft.  
Wellhead pressure—shut in—2000 psig.  
Wellhead temperature—flowing—100 degrees F.  
Gas analysis—3 percent  $H_2S$ , 3 percent  $CO_2$   
Water production—2 bbl/day

**Description of failure:** Hardened steel Bourdon tubes have failed on several occasions.

**Remedial measures:** Satisfactory service has been obtained with Type 316 stainless steel Bourdon tubes.

#### No. 7

Failed item—Hardened steel valve stem.  
(Well location as in No. 6 and well conditions substantially the same)

**Description of failure:** One hardened steel (R. 28) valve stem failed, the crack starting at root of a thread. Failure was characterized as brittle.

**Remedial measures:** K-Monel has given satisfactory service in this case.



## No. 8

Failed item—N-80 casing  
Well location—Ginger Field, Texas  
Depth—12,000 ft.  
Wellhead pressure—shut in—3000 psig.  
Bottom hole temperature—250 degrees F.  
Gas analysis—40 percent  $H_2S$ , 5 percent  $CO_2$   
Water analysis—No sample available  
Material in annulus—None. Completed open ended

**Description of failure:** After 62 hr. operation failure of the  $\frac{5}{8}$ -inch production casing string (N-80, 20 lb.) occurred at 1300 ft. The failure was in or just below the last thread of the joint and was a transverse failure. No metallographic examination has been made of the area of failure, and it cannot be concluded that the failure was sulfide corrosion cracking since there is a possibility that the failure was purely mechanical.

**Remedial measures:** Annulus packed off to protect casing. Well shut in.

## No. 9

Failed item—Type 410 stainless steel valve plugs (5)  
(Well location and well conditions the same as in No. 8)

**Description of failure:** During initial operation of the well five failures of Type 410 stainless steel (12 Cr) plugs occurred in valves on the Christmas tree and at the separator. Failures occurred in 6 to 80 hours operation. In each case the plugs had been flame-hardened to R<sub>c</sub> 40-45, and it is thought that the internal stresses resulting from this treatment were a major contributing factor.

**Remedial measures:** Replacements made with K-Monel plugs. Well shut in, therefore no information available as to whether this measure is satisfactory.

## No. 10

It is known that numerous failures of tubular goods, wire lines, Bourdon tubes, and steel springs have occurred in oil wells in the Worland Field in Wyoming. The gas produced with the oil contains over 30 percent  $H_2S$ . As yet no detailed information on these failures has been made available to the committee.

Despite the limited number of proved instances of sulfide corrosion cracking in the above listing there is good evidence that such an effect exists and that both tubular and wellhead equipment in sour gas condensate wells may be effected. The principal point of similarity in these various failures is that high strength steel alloys were involved; in every case the material that failed had a nominal yield strength above 80,000 psi. The effect was not confined to a particular alloy type, failures being observed with nickel steels, chromium steels and the manganese-molybdenum steels (N-80). In most cases the magnitude of the applied stress at the time of failure was not known; in the one case (No. 1) where an estimate was made, the value obtained was somewhat below the yield strength.

Cracking occurred in wells producing gas containing at least 3 percent hydrogen sulfide, the hydrogen sulfide partial pressure being in the range 60-1200 psia. An exception might be noted in case of failures No. 2 and 3, where cracking apparently started on the external surface of the tubing which was not in contact with the produced fluid but with bunker fuel. Presumably some hydrogen sulfide would be

present in the fuel, although the partial pressure would be quite low.

It is interesting to note that, in general, failures have occurred after relatively short exposure of the failed item to the corrosive environment. This indicates that if no failures should occur with a particular well after the initial operating period, there is only slight probability that failures will occur subsequently, providing, of course, no changes are made which would increase the loading of some susceptible item or would change the corrosiveness of the well fluids.

Since failures occur so rapidly and the nature of the attack precludes there being any warning of imminent failure, inspection of well equipment would seem to have little value as a preventive method.

## Field Tests

Two operating companies are making extensive field tests with stressed specimens of various alloys exposed in well flowlines. In addition Committee TP-1G has conducted a cooperative field test in the McKamie-Patton Field. A summary of the data from these three tests is presented in Table I.

In tests at the Pincher Creek Field failures were observed with several special alloys as well as with 9 percent nickel steel and 12 percent chromium steel, but not with N-80 or J-55 steels of average properties. In the Jumping Pound tests failures were observed with 9 percent nickel steel, 7 percent chromium steel, and SAE 4340 steel, but not with N-80 or J-55.

These two tests cannot be compared directly as different alloy stocks were employed and methods of stressing and exposing specimens varied, but there appears to be a rough similarity in results. These results confirm earlier observation that the effect is associated with high strength steels and that a rough correlation with mechanical properties is possible. In these two tests all materials of hardness greater than Rockwell C 24 failed, and no materials with hardness less than Rockwell C 21 failed. Since so many other factors may enter into the situation, it is suggested that caution should be exercised in the use of hardness alone as a means of establishing susceptibility of a given material. Of particular significance is the fact that the cracking effect can be made to occur with simple test specimens exposed in the well flowline. This provides a useful method for evaluating various materials prior to use. It is expected that such testing would be done in conjunction with a laboratory test program, the latter serving to screen out completely unsatisfactory materials.

That no failures were observed in the test at the McKamie-Patton Field was unexpected. No ready explanation is available although the following points of difference are apparent:

1. Wellhead temperatures in the McKamie-Patton test were somewhat higher than in the other field tests.
2. Composition of the product and water phase has been shown to be a factor in sulfide corrosion cracking and there may be significant differences in salt content or organic acidity that might account for the test results. No comparative analytical data are available at this time.

3. Differences in protective action of the produced hydrocarbon may account for the differences. Comparative wettability tests, such as the drop-size ratio test, should help to clarify this possibility.

It has been reported that weight loss corrosion data obtained during these various tests have indicated only mild general corrosion to occur; corrosion rate values based on total weight loss have been lower than 10 mils per year.

### Preventive Measures

To protect casing the current practice is to set a packer and fill the annulus with some non-corrosive material. For tubing the problem becomes more complex. The measures employed to prevent tubing failures have been some combination of the following:

1. Use of materials with the lowest tensile and yield strengths consistent with the mechanical requirements.
2. Design of the tubing string so as to have the lowest possible unit stresses,
3. Use of protective coatings, and
4. Use of corrosion inhibitors.

For valves and wellhead fittings the above considerations will also apply. In addition use of special alloy linings and special alloy parts should prove feasible. With small equipment items the problem of sulfide corrosion cracking has been met by replacement with Type 316 stainless steel or K-Monel.

From a long range viewpoint it appears that the most desirable solution to the problem would be to develop new high strength tubular materials that

are completely resistant to sulfide corrosion cracking. This will probably involve changes in both alloy composition and heat treatment. Such a development is not expected during the current emergency, but it is hoped that the basic information for the development of such materials can be obtained in the near future. It is for this purpose that the NACE through Committee TP-1G is sponsoring a research project at Yale University under Professor W. D. Robertson. This research project, which was initiated in July, 1951 with funds provided by The International Nickel Company, is concerned with obtaining a fundamental understanding of the metallurgical aspects of sulfide corrosion cracking.

### Acknowledgement

Progress of the committee's work has been made possible by the active cooperation of the individual committee members and the willingness of the companies represented to submit information to the committee.

### Committee Roster

A. C. Broyles, The Carter Oil Company	F. A. Prange, Phillips Petroleum Company
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V. V. Kendall, National Tube Company	R. S. Treseder (Chairman), Shell Development Company
B. B. Morton, The International Nickel Company	L. W. Vollmer, Gulf Research and Development Company
G. R. Olsen, The Pure Oil Company	

TABLE I—Summary of Field Tests of Sulfide Corrosion Cracking

Exposure Conditions:	Pincher Creek	Jumping Pound	McKamie-Patton
Production rate, MMCF/Day	3.0	4.8	6.6-11.4
H <sub>2</sub> S content of gas, percent	10.0	3.5	6.5
CO <sub>2</sub> content of gas, percent	6.0	6.0	2.7
Pressure in test section, psig	2250	2200-2500	1800-2200
Temperature of test section, °F	90	90	165
Duration of specimen exposure	9 months	2-4 weeks	4-6 weeks

Specimens were located in flowline just beyond wellhead.

Test Results (X = failure, O = no failure)

ALLOY	Yield Strength psi	Ultimate Strength psi	Rockwell Hardness C Scale	Prestress (Tensile) psi	Pincher Creek <sup>1</sup>	Jumping Pound <sup>2</sup>	McKAMIE-PATTON	
							Gulf Type <sup>1</sup>	Shell Type <sup>2</sup>
J-55	54,000	92,000	(53A)			O		OO
J-55	60,300	96,300	(95B)	96,300	O			
J-55 H <sub>2</sub> O Quench	115,000	125,000	31	125,000	X			OO
N-80	91,000	118,000	21			OO		
N-80	96,000		23	110,000			O	
N-80	85,000	124,000	26	124,000			O	
N-80	85,200	122,700	24	122,700	O			
9 percent Ni steel	106,000	126,000	24			XXOO		OO
9 percent Ni steel	97,800	116,000	21	98,800	X			
9 percent Ni steel	98,000		22	107,000			O	
9 percent Ni steel	96,000	117,000	23	117,000			O	
5 percent Ni steel	66,000	99,000	(54A)					OO
5 percent Ni steel	61,000	91,000	(52A)			OOOO		
5 percent Ni steel	62,300	97,800	(92B)	63,300	O			
7 percent Cr steel	126,000	158,000	30			X		OO
12 percent Cr steel, cast	55,000	84,000	(50A)			O		OO
12 percent Cr steel, wrought	120,200	161,000	33	121,200	X			
SAE 4340	126,000	148,000	27			XX		
5 percent Ni + Cr steel	115,000		28	125,000			O	
5 percent Ni + Cr steel	114,000	134,000	29	134,000				
5 percent Ni + Cr steel	113,000	133,300	27	114,000	XX			
5 percent Ni + Mo steel	80,000		21	101,000			O	
5 percent Ni + Mo steel	85,000	122,000	22	122,000			O	
5 percent Ni + Mo	83,700	126,000	23	120,600	X			
5 percent Ni + Mo	83,700	120,600	23	84,700	O			
9 percent Ni + Ti steel	92,000		18	98,000			O	
9 percent Ni + Ti steel	92,000	103,000	17	103,000			O	
9 percent Ni + Ti steel	93,100	97,700	20	98,800	O			

1. Gulf type specimens and holders used. Specimens were 0.18 x 0.20 x 10.0 in., stressed as beams with stress applied at the third points to give uniform stressing in the center section. Specimens insulated from holder. Prior to test specimens were prestressed to the ultimate strength or to a stress midway between the yield strength and the ultimate strength. In test the specimens were stressed to a maximum fiber stress of 80 percent of the yield strength.

2. Shell type specimens and holders used. Specimens were 0.06 x 0.25 x 3.0 in. and stressed as a simple beam with stress applied at the center. Specimen insulated from holder. No prestressing prior to test. All specimens stressed to a nominal maximum fiber stress of 120,000 psi. Half of specimens had stress raisers in the form of two No. 60 holes drilled on the centerline near the middle of the specimen.

# Hydrogen Embrittlement Tests On Various Steels \*

By F. A. PRANGE\*

EMBRIITLEMENT failures in steel are not new to the oil industry. They have long been known in the handling of streams containing hydrogen sulfide. With the large scale use of hydrofluoric acid, the same troubles were experienced in this newer medium. Brittle type failures occurred in bottom hole equipment used in sour oil wells, in valve stems used in sour condensate wells, in gasoline plant relief valve springs, in refinery bolting on the inside of vessels, in chemical equipment used for processes utilizing hydrogen sulfide, and in bolting and springs used in hydrofluoric acid alkylation units.<sup>1</sup> The environments causing the brittle failures all have one point in common. At the same time that they cause embrittlement failures in hard steels, they cause blistering in soft steel. While there are exceptions, cracking generally does not occur in steels that blister.

After the first hydrofluoric acid alkylation unit started operation late in 1942, embrittlement and cracking became a problem of considerable interest. The apparent close connection between blistering and cracking led to cathodic pickling experiments to determine blistering and cracking characteristics of steels. Limited tests on hard steels indicated that steels above Rockwell C-20 were susceptible to cracking. This figure coincided with the figure of Rockwell C-20 indicated as the limiting hardness for exposure in hydrofluoric acid by plant experience.

When failures of tubing in sour condensate wells became of interest, the cathodic pickling tests were made on steels that might have use for tubing materials in deep corrosive wells. In devising the test procedure, it was assumed that under field conditions corrosion in the sulfide environment loaded the metal with hydrogen and that the hydrogen in the steel was responsible for the final failure. Because corrosion may vary considerably from piece to piece, it seemed desirable to use conditions that would load specimens with hydrogen while no corrosion or the lowest possible degree of corrosion took place. Cathodic pickling seemed to satisfy this requirement.

Specimens used were U-shaped specimens of steel, heat treated to give varying hardnesses. They were made in this manner: Bars approximately 4 inches

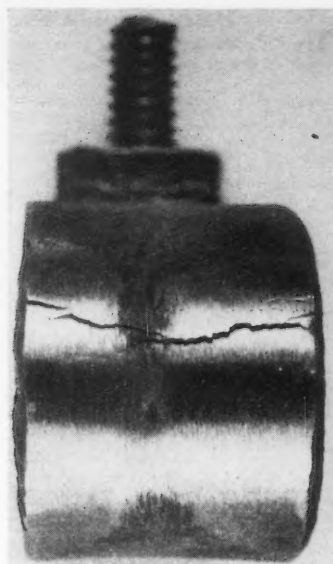


Figure 1—Cracks in cathodically pickled 9 percent nickel steel. 3X.

## Abstract

Cathodic embrittlement tests were made on a number of alloys having good properties for use in deep corrosive wells. The tests suggest that the maximum hardness for use in embrittling environments should be Rockwell C-20.

long, 1/2-inch wide, and 1/8-inch thick were given a 180 degree free bend. Then, these shapes were heated to the hardening temperature, quenched in water or air and then tempered at various temperatures to give the desired hardness. Because the samples were heated to a hardening temperature, there was effective elimination of the cold work of bending the specimens to the U-shape. Before the specimen was placed in the pickling solution, the ends were drawn together slightly with a bolt until yielding was apparent. This resulted in a small amount of cold work, as well as a stress on the material. Scale on the outside radius of the bend was removed with polishing paper before immersion in the solution.

The pickling solution was a 5 percent sulfuric acid. The solution contained 20 milligrams per liter of arsenic added to promote the hydrogenizing of the steel. Temperature of the bath was about 100 degrees F; current density about 2 amperes per square inch. All specimens were exposed 105 minutes, though in some cases visible cracking occurred in as little as 10 minutes. Corrosion, if present at all, was not visible. Indeed, corrosion would not be expected to occur under cathodic pickling conditions in a solution of inhibited sulfuric acid. Thus, the conditions used in this investigation are as close to pure hydrogen embrittlement as seems possible. Results are shown in Tables I, II and III.

Though the test was not quantitative, several degrees of damage are distinguishable. Cracks may

\*A paper presented at the Eighth Annual Conference, National Association of Corrosion Engineers, Galveston, Texas, March 10-14, 1952.

\*Phillips Petroleum Co., Bartlesville, Okla.



**TABLE I—Hydrogen Embrittlement Tests on Various Alloys**  
Samples cathodically pickled to induce hydrogen embrittlement. Electrolyte was 5% SO<sub>4</sub> with 20 mg of arsenic per liter. Current density was 2 amp. per sq. in. Exposure time was 105 minutes.

Material	Hardness As Received (Rockwell)	Hardening Treatment	Hardness After H.T. (Rockwell)	Tempering Treatment*	Tempered Hardness (Rockwell)	Sample Cracked	Relief of Bolt Stress	Remarks
N-80 Tubing	C-28	35 Min.—1475° F Water Quench	C 60	1 Hr.—1120 1 Hr.—1150 1 Hr.—1200 1 Hr.—1250	C 29 C 25 C 20 B 97 (C 17)	Yes Yes Yes Yes	Yes Yes Yes Yes	Blistered
3½% Ni	B 83	35 Min.—1475° F Water Quench	C 32	None 1 Hr.—800 1 Hr.—950 1 Hr.—1200	C 32 C 26 C 19 B 94 (C 11)	Yes Yes Yes No	Yes Yes Yes No	**
5% Ni	B 86	35 Min.—1475° F Water Quench	C 38	1 Hr.—600 1 Hr.—800 1 Hr.—950 1 Hr.—1200	C 31 C 26 C 20 B 93 (C 9)	Yes Yes Yes No	Yes Yes Yes No	
9% Cr	B 92	35 Min.—1700° F Air Quench	C 40	1 Hr.—1120 1 Hr.—1150 1 Hr.—1200 1 Hr.—1250	C 28 C 24 C 21 B 97 (C 18)	Yes Yes Yes No	No No No No	
12% Cr	B 88	35 Min.—1700° F Air Quench	C 32	None 1 Hr.—1050 1 Hr.—1120 1 Hr.—1200	C 32 C 25 C 19 B 96 (C-15)	Yes Yes V. Slight No	Yes Yes No No	
AISI 4140 Steel	B 96	35 Min.—1475° F	C 41	1 Hr.—950 1 Hr.—1020 1 Hr.—1120 1 Hr.—1200	C 32 C 25 C 20 B 95 (C 14)	Yes Yes No No	Yes Yes No No	2 Samples. Same Result
Low Carbon Skelp	B 82	None		None	B 82	No	No	

\* Air Quenched. \*\* Spalled at Crack

be so slight that they are barely visible. Other cracks may be deep. Finally, cracking may be so deep that the bolt holding the ends of the "U" together may be completely unloaded. This latter degree is indicated under the column marked "Relief of Bolt Stress" in the tabulations of the results. Figure 1 shows typical fairly severe cracking. Blisters were formed on only one sample; however, the cathodic pickling process used in these tests is capable of causing very rapid blistering on soft, dirty steels.

The nature of the cracking varied from one alloy to another. In 12 percent Cr steel and 4140 steel, the cracking was largely transgranular. In 9 percent Ni steels, it seemed to be intergranular; although in some cases etching procedures did not adequately delineate grain boundaries for certain identification. Figure 2 is a photomicrograph of cracking in a 9 percent nickel steel sub that

**TABLE II—Hydrogen Embrittlement Tests on 9% Nickel Steel**

MATERIAL	Hardening Treatment	Hardness After H.T. Rockwell	Tempering Treatment	Tempered Hardness Rockwell	Sample Cracked	Relief of Bolt Stress	Remarks
9% Nickel Tubing.....	None	....	None	C 26	Yes	Yes	
Rockwell C 26.....	None	....	None	C 26	No	....	Not stressed in test
As Received.....	None	....	None	C 26	No	....	Cold Worked, but not stressed in test
	25 Min.—1470° F Air Quench	C 35	2 Hr.—950° Air Q.	C 28	Yes	Yes	
	25 Min.—1470° F Air Quench	C 35	2 Hr.—1050° Air Q.	C 24	Yes	Yes	
	25 Min.—1470° F Air Quench	C 35	1 Hr.—1100° Furnace Cooled	C 20	Yes	Yes	
9% Nickel Tubing From Failed Sub. Rockwell C 24 as Received	None	....	None	C 24	Yes	Yes	Not Stress Relieved
9% Nickel Sucker Rod.....	None	....	None	B 17 (C-17)	No	No	Not Stress Relieved
Rockwell B 97 as received	None	B 95	None	B 95	No	No	
9% Nickel Strip, Rockwell B 95..... as received	1575 F Air Cool	C 26	None	C 26	Yes	Yes	
	None	....	None	C 26	No	....	Cold worked, but not stressed in test

**TABLE III—Hydrogen Embrittlement Tests on Special Nickel Steels**

Material	Hardening Treatment	Hardness After H.T. Rockwell	Tempering Treatment	Tempered Hardness Rockwell	Sample Cracked	Relief of Bolt Stress
5% Ni-Mo	As Received 1 Hr.—1650° F Air Quench	C 32	As Received None 35 Min. 1050° Air Q. 35 Min. 1200° Air Q. 3 Hrs. 1200° F.C.	C 23 C 32 C 24 C 20 B 96 (C-17)	Yes Yes Yes Yes No	Yes Yes Yes Yes No
5% Ni-Cr	As Received 1 Hr.—1650° F Air Quench	C 17	As Received 1 Hr. 900° Air Q. 35 Min. 1050° Air Q. 35 Min. 1200° Air Q. 3 Hrs. 1200° F.C.	C 27 C 32 C 26 C 20 B 98 (C-18)	Yes Yes Yes Yes No	Yes Yes Yes Yes No
9% Ni-Ti	As Received 1 Hr.—1575° F Water Quench 1 Hr.—1575° F Water Quench	C 24 C 24	As Received 5 Min. liq. nitrogen None 1 Hr. 1050° Air Q. 3 Hrs. 1050° Air Q.	B 96 (C-17) C 26.5 C 24 C 21 B 97 (C-17)	No Slight Slight No No	No No* No* No No

\* Remarks: Very shallow crack.

failed in service in a sour condensate well. Figure 3 shows that the cracking produced by cathodic pickling of 9 percent nickel steel resembles the cracking found in service.

Table III shows the results on special nickel steels proposed for use in tubing. The properties of these special steels are shown in Table IV. The 9 percent nickel-titanium steel showed the best resistance of any steel tested, having only very slight cracking at a hardness of Rockwell C-24. Presence of

residual austenite in the 9 percent nickel-titanium was demonstrated by the liquid nitrogen treatment, which increased the hardness from Rockwell C-24 to Rockwell C-26½. Despite the residual austenite, this steel was still very resistant.

In summary, composition seems to affect the resistance to cracking only very slightly. The limiting hardness for crack-free operation under severe hydrogen embrittlement conditions seems to be about Rockwell C-20. In the hardness range tested, external stress had to be applied to cause failure. Mere cold work or internal stress from heat treating operations seems to be without effect.

The assistance of Mr. V. L. Hayes in conducting this investigation is gratefully acknowledged.

### Reference

- I. M. H. Bartz and C. E. Rawlins. Effects of Hydrogen Generated by Corrosion of Steel. *Corrosion*, 4, No. 5, 187-206 (1948) May.

TABLE IV—Properties of Special Alloys of Table III  
Analysis, heat treatment, and physical properties of these special alloys as determined by the manufacturer were the following:

Material	5% Ni-Mo	5% Ni-Cr	9% Ni-Ti
Chemical Analysis:			
Carbon.....	0.20	0.18	0.078
Manganese.....	.43	.41	.30
Sulfur.....	.012	.019	.023
Phosphorus.....	.013	.010	.013
Silicon.....	.26	.26	.22
Nickel.....	5.06	4.92	9.04
Chromium.....	.03	1.00	.13
Molybdenum.....	.24	.01	.05
Titanium.....	.....	.....	.30
Heat Treatment:			
	Normalize 1650° F	Normalize 1650° F, Temper 1050° F—30 Min.	1575° F Water Quench, Temper 1050° F—3 Hrs.
Mechanical Properties: (Full Section Samples)			
Tensile Strength, psi	123,000	132,000	103,000
Yield Strength, psi	84,000	107,000	88,800
Elongation, % in 2"	38	27	32

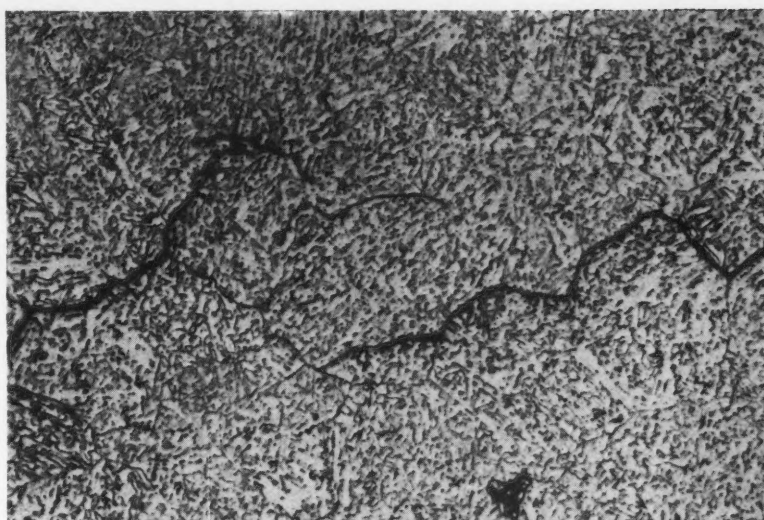


Figure 2—Cracking in a 9 percent nickel tubing sub which failed in a sour condensate well after a few hours' service. Amyl alcohol—nitric acid etch. 200 X.

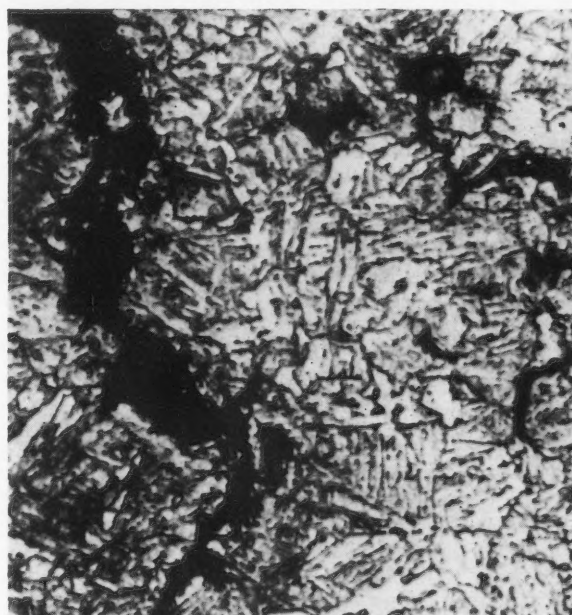


Figure 3—Cracking on 9 percent nickel steel by cathodic pickling. Nital etch. 200 X.

## Discussions on Sulfide Stress Corrosion Symposium: \*

Discussion by John W. Juppenlatz, Lebanon Steel Foundry, Lebanon, Pa.:

The four papers that have been presented with regard to hydrogen sulfide embrittlement indicate the scope of the problem and a review of experimental work that has been done to promote a better understanding of conditions which effect failure.

Mr. Treseder has indicated the conditions that have existed in three wells, where two failures have occurred, while the McKamie-Patton well has operated in a satisfactory fashion. This well has an

operating temperature of 165 degrees F and this temperature may be sufficient to expel any hydrogen embrittlement upon its attack and absorption at, and near, the surface. The effects of temperature should not be overlooked.

The fundamentals of hydrogen embrittlement in hardened steel or other metallic materials is certainly not new. The papers that have been presented show particularly that materials of high hardness levels and under high stress are subject to early failure by embrittlement under conditions of hydrogen sulfide as employed. I did obtain a reaction that most of the materials that have been under discussion were

\*EDITOR'S NOTE: Discussions hereunder pertain to the four articles immediately preceding.

generally unsatisfactory for this type of hydrogen sulfide service, but this is not the case, since there are many installations in operation today where no failures have occurred. Undoubtedly, there are materials with high strength levels that will serve better than other materials, from both resistance to corrosion and hydrogen absorption viewpoints.

The micro-structural conditions have been discussed, and it appears that untempered martensite, regardless of alloy content, will rapidly fail under high stress conditions. Therefore, material selection must be made utilizing certain limiting factors. Materials with excessively high hardness or strength levels apparently must be avoided, along with stresses which exceed the yield point. Engineering structures are usually designed to work under the yield point, so many materials should operate satisfactorily under these hydrogen sulfide well conditions over long periods of time.

If proper application is made, which has been done in the past, we do know that certain alloyed materials have withstood both the corrosive and embrittling effects of these types of hydrogen sulphide service.

#### Reply by R. S. Treseder:

There is insufficient data available to allow the conclusion that the absence of failures during the McKamie-Patton Field tests could be attributed to the higher flow-line temperature. Other important factors such as the low amount of water produced and the wettability of the produced hydrocarbon would have to be considered in any explanation of the McKamie-Patton results. The suggestion that elevated temperature alone will eliminate failures of this type is opposed by field experience in which cracking of 9 percent nickel steel tubing subs occurred at temperatures of the order of 150 degrees F (TP-1G paper, field experience items No. 2 and 3).

#### Discussion by B. B. Morton, The International Nickel Co., Inc., New York, N. Y.:

Some work along the lines discussed in this symposium has been carried out in the laboratory of The International Nickel Company at Bayonne, New Jersey by Dr. H. R. Copson and C. F. Cheng.

The authors used specimens of various steels formed into U-bends with the load maintained on the legs by means of through bolts of Monel. The specimens were immersed in water, saturated with a 50/50 mixture of hydrogen sulfide and carbon dioxide. Conclusions drawn from these tests are:

1. Cracking occurred rapidly under these test conditions. All steels tested cracked. Among those tested were 9 percent nickel, 5 percent nickel, 3 percent nickel, API N-80, 9 percent chromium, 5 percent chromium, in various conditions of heat treatment at various hardness levels.

(The authors did not appear to find any hardness level below which cracking would not occur. This is in contrast to a viewpoint that possibly C-20 hardness represents a level below which cracking would not be expected. The authors also found that no heat treatment was adequate to prevent cracking of the 9 percent nickel steel, which contrasts with the

thought expressed in other quarters that the micro-structure may affect the tendency toward cracking. They also concluded that variations in composition, involving carbon, titanium, molybdenum and chromium, had no major effect.)

2. Damage to the steel by occluded hydrogen was the most important factor in the cracking. The hydrogen produced deep fissures, blisters and embrittlement.

3. The authors conclude that corrosion was necessary to form nascent hydrogen and hydrogen sulfide was necessary to catalyze penetration of the hydrogen into the steel. High tensile stresses were necessary for failure and corrosion may have concentrated the stresses by forming notches.

4. Cathodic protection accelerated rather than prevented failure; similarly, galvanic contact with less noble metals would be expected to be harmful.

(This result is in line with ones previously reported by Fred Prange in which he showed that cathodic protected metal would be subject to cracking in an environment favorable to the introduction of hydrogen into the metal. In his case, arsenic was used as a catalyst.)

5. Nickel plating prevents the cracking, probably by decreasing the corrosion and, accordingly, the penetration of hydrogen. Nickel plated U-bends cracked if charged cathodically with hydrogen.

6. A formaldehyde inhibitor decreased the corrosion of 9 percent nickel steel and API N-80 and prevented the cracking of these steels. This inhibitor did not work in the case of the chromium steels.

(It is noteworthy that the authors, Copson and Cheng, point out that the hydrogen sulfide is responsible for the cracking and that carbon dioxide is not corrosive nor is it essential. They state, however, that the presence of the carbon dioxide would lower the pH value and increase the corrosion rate, especially under high pressures.)

#### Reply by J. P. Fraser:

Mr. Morton, together with co-workers Copson and Cheng, have concluded that occluded hydrogen is responsible for sulfide corrosion cracking failures; and they have found that cathodic protection accelerated failure of susceptible materials in their tests. In contradiction to this are results from several experiments made in our laboratories on the effects of cathodic protection under sulfide corrosion cracking conditions. In these experiments, stressed specimens of 9 percent nickel steel were exposed to several different environments with the simultaneous application of a cathodic current. The experimental results were as follows:

1. Cracking was prevented at moderate current densities in distilled water saturated with hydrogen sulfide.
2. Cracking could not be prevented by cathodic currents in a 0.5 percent acetic acid solution saturated with hydrogen sulfide, even at very high current densities.
3. It was found to be impossible to produce cracking in a 0.1 M borax solution, even if arsenic were added as a "promoter," at these stress levels. With cold-worked specimens (stressed U-bend), it was possible to crack the 9 percent nickel steel with cathodic hydrogen in the 0.1 M borax solution even in the absence of arsenic.



Our interpretation of these data is that stress corrosion must be the predominant mechanism in sulfide corrosion cracking. If it were not, then cracking would have been expected in the borax solution, since the specimens were as embrittled due to occluded hydrogen under these conditions as on exposure to distilled water saturated with hydrogen sulfide in the absence of impressed currents. Lack of protection in the dilute acetic acid solutions may be ascribed to the imperfect protection provided by the cathodic currents, since a small but appreciable amount of corrosion still took place even at the highest current densities used.

**Discussion by W. D. Robertson, Yale University, Cambridge, Mass.:**

The extensive work of Bowers, McGuire and Wiehe, Fraser and Treseder and Prange, indicates that certain generalizations may be made regarding the mechanism of sulfide cracking.

1. It appears to be associated with metallurgically unstable alloys; that is, alloys which are unstable with respect to internal stress and strain induced precipitation. Unfortunately the conditions that lead to the required higher strength seem to be those resulting in susceptibility to failure in hydrogen sulfide.
2. The character of both laboratory and service failures is that of brittle fracture indicating that materials exposed to  $H_2S$  under stress are unable to flow and redistribute local stress concentrations with the result that stress increases until a single microscopic fracture occurs, probably beginning at some point of stress concentration.
3. Failure results from *simultaneous* exposure to the environment and applied or internal stress.
4. Failure in environments containing hydrogen sulfide has all the characteristics associated with hydrogen embrittlement.

Specifically, Bowers, McGuire and Wiehe have shown the presence of martensite in 9 percent Ni alloys. Since the properties of martensite with respect to internal stress and hardness depend primarily on carbon content of the original austenite it is understandable and significant that the addition of titanium should result in a more stable alloy which is less susceptible to cracking. A martensite network is highly improbable and if present would result in brittleness in the absence of any environment.

The coating experiments of Fraser and Treseder may be a demonstration of the requirement that simultaneous exposure and stress are necessary for failure at the point of exposure which is in accord with the fact that hydrogen primarily affects the ductility of iron as measured in the absence of the environment. Extensive measurements of mechanical properties made in the environment are required to advance our understanding of this aspect of the problem. The effect of temperature shown by Fraser and Treseder is in the expected direction for a process involving  $H_2$  embrittlement but should be extended to include a determination of the quantity of hydrogen actually in the specimen at different temperatures.

Prange's experiments with 9 percent Ni alloys containing titanium also demonstrates its stabilizing effect. In an alloy containing about 0.1 percent C and 0.3 percent Ti the function of the latter cannot be other than one of reducing the effective carbon content. It would be very instructive to lower the carbon content below 0.001 percent by other means and to determine whether the susceptibility to cracking is similar to the titanium containing alloys. The correlation of blistering in low alloy steel and absence of blistering accompanied with susceptibility to failure in high strength, high alloy steels may simply be an expression of the fact that the stress level in a low alloy steel cannot increase sufficiently to cause brittle fracture whereas the higher strength alloys are unable to relieve local stress concentration by plastic flow and consequently brittle fracture may take place, essentially under conditions of triaxial stress.

In summary it appears that the solution to the problem requires more definite knowledge of the following factors.

1. The effect of hydrogen on the plastic deformation of iron.
2. The possible role of hydrogen absorption in causing a state of internal stress, essentially triaxial, which in low alloy steel results in local blistering and in high strength alloys may result in the necessary stress state for brittle fracture.
3. The role of carbon which, associated with stress, may result in phenomena analogous with strain ageing and other reactions characteristic of metastable alloys.
4. The role of impurities like phosphorus which are both promoters of  $H_2$  absorption and are known to effect the fracture characteristics of ferrous materials.

**Reply by J. P. Fraser:**

Professor Robertson observes that sulfide corrosion cracking "appears to be associated with metallurgically unstable alloys." Since no steel is completely stable from a thermodynamic point of view, this immediately raises the question of what constitutes a "stable" alloy. Of interest in this connection is an experiment in which a sample of SAE 4340 steel was hardened from 1630 degrees F and tempered for one hour at 1280 degrees F, placing it in a fairly soft and relatively stable condition. Stressed specimens of this material failed in our test. It appears that even a material which is, for most practical purposes, metallurgically stable can be susceptible to sulfide corrosion cracking.

Professor Robertson also states that failure in environments containing hydrogen sulfide (i.e., sulfide corrosion cracking) has all of the characteristics associated with hydrogen embrittlement. This is not in accord with the observation that many materials which have appreciable ductility when "hydrogen embrittled" fail with no apparent plastic deformation on exposure to sulfide corrosion cracking test conditions.

**Discussion by Robert T. Effinger, Shell Oil Co., Martinez Refinery, Martinez, Cal.:**

There are many features of the problem of embrittlement of high strength steels in hydrogen sulfide environments which are common with the problem of blistering of low carbon steel used for the con-

struction of pressure vessels and the like. Steels become embrittled, blistered, and fissured as a result of penetration by hydrogen, and the environments which promote this hydrogen penetration into steel can cause any one or all of these damaging effects.

One might conclude from the tenor of these papers and discussions that only the high strength steels lose ductility in the environments promoting hydrogen penetration. Refinery experiences indicate that low carbon steels also lose most of their ductility when charged with hydrogen. In fact, low carbon steel such as ASTM A-285 Grade C and ASTM A-201 when charged with hydrogen may have an elongation in the order of 1-10 percent when tested in tension or by slow bend. Also fissuring of steel occurs in these environments and the fissures are normally aligned perpendicular to the major stresses in the steel and thus have a maximum damaging effect.

I believe it can be concluded that any carbon or low alloy steel exposed to environments which are strongly active in promoting hydrogen penetration into the steel will suffer serious damage by blistering, fissuring and embrittlement. Much has been done to investigate these effects upon the metal and the evidence indicates that all ferritic steels suffer from the damaging effects of hydrogen. The tendency for the high strength steels to embrittle and break in corrosive sulfide environments may be alleviated slightly by softening the temper of the steel. This has the attendant disadvantage that tensile strength is lowered and thus in part, defeats the original purpose for using high strength steel.

Very little is known about the factors in the environment which influence the corrosion reactions responsible for hydrogen penetration. It appears to me that we might profitably investigate the chemical factors of the environments with the aim of controlling the corrosion reaction by inhibition or other treatments and thereby prevent hydrogen penetration into steel. If this aim can be realized we can then make full use of the strength and other desirable physical properties of the ferritic steels.

**Question by E. W. Wallace, 5304 Huisache, Bellaire, Texas:**

Please describe etching technique used by Mr. Bowers.

**Reply by C. N. Bowers:**

The structures were developed by etching in an equal mixture of 2 percent Nital and 4 percent Picral.

**Reply to Discussions by C. N. Bowers, W. J. McGuire and A. E. Wiehe:**

We agree with the results reported by Mr. Morton which show that any material can be made to fail under the selected environment if subjected to suf-

ficient cold working and/or stressing. However, we know some of the materials tested will perform satisfactorily under sulfide conditions. For this reason, laboratory test specimens should be conditioned and stressed to levels that will not be exceeded in service.

Dr. Robertson's discussion gives the impression that the problem is solely one of embrittlement caused by absorption of hydrogen. Mr. Juppenlatz also stated that the papers presented show that materials at high hardness levels and under high stressing are subject to early failure by embrittlement under conditions of hydrogen sulfide. Our work, at least, does not show that laboratory or service failures were due to embrittlement alone and we do not, for the present at least, consider embrittlement a primary cause of failure, although it may be an important contributory factor.

Steels that are susceptible to failure, with or without plastic deformation, will not fail unless the applied stress is above a "critical" amount. However, the stresses in oilwell tubing, for example, are quite high in deep, high-pressure wells. For these wells the problem of supplying tubing having sufficient mechanical properties from a standpoint of good engineering design and yet being free of damaging martensite in the structure and plastic deformation due to cold straightening is certainly not quite as simple as indicated by Mr. Juppenlatz.

#### **General Discussion by R. S. Treseder:**

Some of the discussion has been concerned with the detailed differences obtained by the various authors under their special laboratory conditions. It should be pointed out that the general results obtained in these investigations are remarkably similar and that the general conclusions as regards possible practical solutions to the problem are almost identical. Under a variety of test conditions it has been shown that hardened steels are subject to sulfide corrosion cracking, the susceptibility becoming quite apparent at hardness values of the order Rockwell C20 and higher. Unless conditions permit use of special non-ferrous alloys, it is generally agreed that a material of minimum hardness compatible with use requirements should be employed and an effort made to minimize residual and applied stresses.

It should be noted also that this symposium is somewhat unusual in that an important industrial corrosion problem is being discussed in detail at an early stage before satisfactory solutions to the problem have been established. The sulfide corrosion cracking problem is being presented to industry now for the purpose of 1) providing a warning to those companies who have not experienced this type of failure, and 2) provoking the interest and suggestions of those not directly concerned with the problem.

# Corrosion Control by Magic—It's Wonderful\*

By H. H. UHLIG\*

CONTROLLING corrosion by sleight of hand and by magic has much to recommend it. The first cost is not unreasonable, and the upkeep is simple. Often, one merely plugs the device into a convenient socket, or by-passes a portion of the water main, thereafter forgetting everything, including the works chemist whose services are no longer required. Like as not, the magic device throws in, free of charge, scale removal, deodorizing, algae extermination and water softening.

But like the Scotchman who happily succeeded in getting his horse to eat but one straw a day, only to have the horse die, these corrosion control devices suffer from the one defect that after an initial period of brilliant success, the boiler, or condenser, or piping disgustingly succumb to a malady strongly resembling corrosion. Promoters who sell this magic as a reasonable facsimile of science usually fall into two categories illustrated by the following accounts.

In 1903, Professor Blondlot at the University of Nancy, France, announced his discovery of the new and mysterious N rays. These rays were emitted by metals, living animal tissue and growing plants, including vegetables. One hundred papers were published in *Comptes rendus* describing properties of the rays, and even how their wave length could be measured. The French Academy of Science announced the award of a gold medal and 20,000 francs to Blondlot in recognition of this discovery.

There was only one hitch. Few investigators could observe the phenomena by repeating the described experiments. French Academy or no, the subject was embarrassing to scientists in other countries who tried hard to find the N rays but never succeeded. Professor Rubens of Berlin was distressed by a command from the Kaiser to demonstrate the N rays at Potsdam, which resulted only in his forced admission that he was unable to do so. Englishmen, too, were threatened with an inferiority complex. Something had to be done.

In the summer of 1904, at a meeting of the British Association for the Advancement of Science, disillusioned scientists cornered an American professor of physics, R. W. Wood, from Johns Hopkins University. He had a reputation for being able to see through fuzzy ideas and scientific high jinks. At first he demurred, but later agreed to take the trip to Nancy. Professor Blondlot gladly repeated the experiments that had made him famous. But the visual effects he indicated were not all apparent to Wood. The final experiment was one in which Professor

## Abstract

The author, citing an historic case of self-delusion and another instance of an outright attempt at fraud in the promotion of claims to scientific discoveries, suggests that lack of rapid evaluation technique and defective education in engineering are responsible in part for the ease with which bogus "dopes" and processes are sold. While urging that new ideas not be discounted in advance it is urged that claims for new ideas and devices be checked by known scientific methods before acceptance.

The following guides are suggested for evaluating new schemes for controlling corrosion: 1) If claims are based on testimonials and not on available authoritative data, dismiss the device until authoritative evidence is supplied. 2) Be cautious concerning devices claims for which include statements obviously wrong. 3) Be cautious of devices operated by no known scientific principles.

The National Association of Corrosion Engineers is advised to set up a council of respected members of the corrosion engineering profession to pass on acceptable advertising in Corrosion and acceptable exhibits at its national conferences.

Blondlot read off wave lengths using a special spectroscope which included an exposed aluminum prism. The room was darkened and the Professor had no trouble reading off several numbers on a graduated scale. Wood asked if he would repeat the experiment, to which Blondlot cheerfully complied. In the subdued light, Wood sneaked over and placed the aluminum prism in his pocket, noting, nevertheless, that the N ray expert read exactly the same lines as before. This convinced Wood he was dealing with a self-deluded individual who had hypnotized himself into believing he actually saw things that did not exist. Next morning, he sent a letter to *Nature* describing his experience. The scientific world was in turmoil for several weeks following, but the upshot was that no more papers appeared on N rays, and the subject plus its inventor passed into obscurity.

Dr. Wood always distinguished Professor Blondlot's fiasco from others, where the intentions of the chief promoter could not be labelled self-delusion, but instead plain and simple fraud. One instance concerned an inventor who was trying to interest American capital in obtaining free power from the atmosphere. He set up a demonstration in a metropolitan hotel to which reporters and financial backers were invited. Dr. Wood attended, noticing a table in the room covered with toy trains, motors and other electrical gadgets. At the end of the table there was a pole from which several brass points radiated. This, it was said, gathered electricity from the atmosphere and supplied the necessary power. It was wonderful and convincing, but not to Dr. Wood. He characteristically looked under the table and noticed several covered boxes, but with no apparent connection to the

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apparatus above. He pulled the burlap from one and found, as he suspected, a good sized storage battery from which wires ran up the center of a table leg. The "inventor" cleared out of the hotel without a porter to carry his hurriedly packed equipment, and without paying his bill.

These two extremes of questionable promoters operate in practically all fields of human activity. One is a respectable, perhaps educated individual, more or less twisted mentally, well versed in scientific language, but with self-deluded thoughts and proposals that have no necessary connection with reality. The other extreme is the slick operator, or plain fraud, who hopes to fascinate someone or some industry with his idea, collect a down payment, and get out before the fraud is exposed. There are all degrees of proposals and intrigues between these two extremes.

The hocus-pocus devices sold by persons in these categories range from proprietary medicines and electric belts to cure rheumatism and cancer, to carburetors that "double" gasoline mileage, and "dopes" that revive automobile storage batteries, or small tubes that shaken in the vicinity of a boiler give off radiation that prevents scaling. In the field of corrosion, there are similarly many gadgets offered and sold that operate by no known scientific principles, and which, so far as the evidence goes, have never been subject to even elementary engineering tests. Some of these, charitably interpreted, accomplish perhaps part of the exaggerated claims made for them. The fact that these "Brooklyn Bridges" are sold every day by strangers to engineers and purchasing agents of reputable companies indicate scientific illiteracy on an amazing scale, and is, in part, a reflection on the type of technical education we are providing those who naively buy such equipment. Perhaps this situation is one of the strongest arguments for better schooling of our engineers in the subject of corrosion on par with present instruction in mechanical properties of metals. Adequate courses in corrosion are offered today at perhaps less than a half dozen technical schools and universities in the country. The National Association of Corrosion Engineers-sponsored short courses have been a help. Considering everything, however, it is small wonder that the young engineer or chemist face to face with the urgency of his first corrosion problem has only hazy ideas of what to do, and falls easy prey to the "fortune teller" and bogus salesman.

It would be a mistake, of course, to instill the notion that all new ideas should be relegated to the category of fakes, and that all proposals should be thrown out that deviate in some degree from what has been done before. Subscribing to such a policy—an easy swing of the pendulum away from the gullible customer mentioned above—would inflict undue penalties on the legitimate inventor. A sounder viewpoint is to critically examine and check claims for new ideas and devices—not by hearsay, nor by the salesman's word for it—but by results of bonafide engineering and scientific tests supplemented by laboratory investigations. Since such tests are often not possible for the individual, or small company, per-

haps the professional societies whose interests overlap corrosion might well consider initiating and supervising a certain number of cooperative tests of this kind in order to help keep their members informed. They could then more reliably scrutinize advertising published under their auspices, or equipment offered for exhibit at national conventions.

In any event, scientific and engineering tests are the final criteria—not the appearance of pipes, the visual thickness of rust films, the lack of suspended rust, or the color of corrosion products. There must be adequate metal thickness checks, weight loss determinations and chemical analyses conducted in tests with adequate control of metal surface preparation, temperature, water composition, aeration, velocity and pH.

The following guide may be of use in a preliminary sorting out of the chaff from the wheat in dealing with new schemes of this kind.

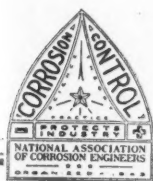
1. Beware of testimonials. Pink pills, electric belts and perpetual motion are all sold on the basis of verified letters from satisfied customers. If the proposed device is not based on engineering experiments, the data for which are available and preferably published in a reputable technical journal, dismiss it until such evidence is supplied.
2. Treat with caution any device that includes in its advertising literature statements obviously wrong. Sometimes claims are made that the device conditions water to pH 7, which is "the least corrosive pH" (?), or electrically charges dissolved salts in water systems, or neutralizes excess oxygen by a carbonizer (?). Statements of this kind are your cue that the manufacturer does not know much about the field he is allegedly serving.
3. Similarly treat with caution devices that operate by no known scientific principles. The salesman says he doesn't know how his gadget works, but he is sure it does. These include devices operating by mysterious principles or which guarantee to prevent rusting inside a pipe line by flowing current along the outside, or which soften water or reduce its corrosivity, but without any detectable change in dissolved salts and oxygen by chemical analyses. An exposure of advertising claims for certain of these gadgets is contained in the July issue of the *Journal of the American Water Works Association*, Volume 44, 576-582 (1952).

The science of corrosion has made appreciable gains within the last ten years. Progress has unfortunately been retarded by the superstitious, by fakers and by deluded individuals, all of whom have had rich pickings in this field over the past fifty years. Like the field of boiler water treatment, corrosion lacks rapid evaluation techniques, thereby making it easier to sell phony ideas and devices which exploit the superstition, ignorance, and lack of confidence among those responsible for the operation of boilers, heat exchangers, condensers and similar expensive equipment.

But the science of corrosion has matured, and it is now the right time to support whatever measures are necessary to insure the continued dominance of science over art and guesswork. NACE was wisely organized to advance the field of corrosion, meaning the promotion of science and technology, and to protect the interests of the corrosion engineer. What we have gained should not be compromised by silently watching questionable promoters boring from within, or by unofficially accepting into the Association questionable schemes for corrosion control because of the "income," or because the promoter might be offended if he were told to sell his magic elsewhere. NACE might well consider a council of respected members of the corrosion engineering pro-

fession to pass on acceptable advertising in *Corrosion* and acceptable on exhibits at national conventions similar to councils set up by the American Medical Association and American Dental Association. By so doing, our objectives and intentions are clearly on record and, at the same time, the Association better serves the membership and the Country.

Further progress can be made by all of us working together to weed out superstition, witchcraft, dishonesty, and reactionary ideas that have operated in a direction opposite to the objectives of the Association for so long. In this way, we meet our responsibilities, and we support and insure the status of the corrosion engineer among the recognized engineering professions.



## Discussions

### A Case History Involving Intergranular Failure of Stainless Steel Heater Tubes. By G. A. Works, *Corrosion*, Vol. 8, No. 6, 217-221 (1952) June.

Discussion by Henry Paige, Philadelphia, Pa.:

Mr. Works has ably presented an important corrosion phenomenon encountered in the use of stainless steels and a practical solution to the problem of sensitization. However, to fully understand the basic mechanisms involved, the following metallurgical features should be emphasized.

As pointed out by the author, when sensitization occurs at moderate temperatures examination of the structure shows the presence of a carbide network at the grain boundaries and resistance to corrosion is diminished. But the carbide particles *per se* are not the cause of this diminished resistance. Rather, it is due to the fact that when chromium carbide is precipitated at the boundary, a zone depleted of alloy content is necessarily left adjacent to the particles.

Using the concepts of Uhlig,<sup>1</sup> it is then possible to calculate the minimum amount of chromium necessary in solid solution to preserve passivity. On the basis that one atom of Mo or Cr can passivate 5 atoms of Fe, while 15 percent Mo is necessary to passivate 85 percent Ni, the following results are obtained for the steel composition presented by Mr. Works:

Molybdenum to passivate nickel

$$13.62 \times \frac{15}{85} = 2.41\%$$

Iron passivated by molybdenum

$$(2.95 - 2.41) \times \frac{5 \times 55.84}{1 \times 96} = 1.57\%$$

Chromium required to passivate balance of the iron

$$(67.22 - 1.57) \times \frac{1 \times 52}{5 \times 55.84} = 12.25\%$$

Thus while the center of the grain has the nominal composition, the outer area is below 12.25 percent chromium and does not consist of "stainless" metal.

Under corrosive conditions an electrolytic cell can then be set up in which the depleted zone is anodic to the center and dissolves rapidly.

Consequently, the stabilization treatment at 1600 degrees F has a more important function than to precipitate carbides at the grain boundary and cause them to nodulize.

Assuming that the carbon is completely precipitated as  $\text{Cr}_3\text{C}$  the maximum amount of chromium removed is:

$$.11 \frac{3 \times 52}{1 \times 12} = 1.43\%$$

This leaves the average chromium composition at 14.67 percent.

Therefore the temperature, while not sufficient to redissolve the chromium carbides, causes chromium to diffuse from areas of 16.1 percent to depleted ones until the gradient is minimized and the composition from the center to the edge of each grain is approximately 14.7 percent. This eliminates the galvanic potential and restores a stainless composition to the entire structure.

#### Reference

1. H. H. Uhlig, Passivity in Copper-Nickel and Molybdenum-Nickel-Iron Alloys, a paper presented at the 85th General Meeting of The Electrochemical Society, April 15, 1944.

### Corrosion Resisting Characteristics of Iron Modified 90:10 Cupro Nickel Alloy by W. C. Stewart and F. L. LaQue. *Corrosion*, Vol. 8, No. 8, 259-277 (1952) Aug.

Question by C. M. Jackson, Devoe & Reynolds Co., Louisville, Ky.:

Is the oxide film or scale formed in the heat treating operation cathodic to the base alloy and what is its effect on the anti-fouling action of the alloy?

**Authors' Reply:**

In reply to the question raised by Mr. Jackson, observations of corrosion of surfaces only partially covered by scales formed at high temperatures have shown no evidence that the scale is cathodic to the bare alloy. There has been no opportunity to observe the anti-fouling characteristics of scale covered surfaces. The expectation would be that they would be more resistant to corrosion and, therefore, more likely to foul than scale free surfaces.

**Question by Norman Hackerman, University of Texas, Austin:**

Why are there no local effects beneath the  $\text{Fe}_3\text{O}_4$  found scattered on specimens containing more Fe than can be kept in solution in the 10 percent cupro-nickel?

**Authors' Reply:**

In reply to Dr. Hackerman's question re the absence of localized corrosion beneath the corrosion product films that contain  $\text{Fe}_3\text{O}_4$ , about all that can be done is confirm the observations and draw some inferences from them. These are:

1. Where such non-adherent black magnetic corrosion product films develop, no corrosion advantage is derived from the extra iron content associated with these films. These films, therefore, must be superimposed on adherent and more protective films that lie beneath them and represent secondary reactions of the iron in the corrosion products that diffuse through the rate controlling films.
2. The distribution of the iron rich phase must be in the form of minute particles so uniformly dispersed as not to lead to any localization of corrosion.

3. The black film which contains  $\text{Fe}_3\text{O}_4$  while not protective, is apparently no more noble than the alloy with its normal film beneath it and, therefore, does not cause accelerated corrosion of the underlying alloy. Potential measurements have shown no significant difference in potential in alloys with and without the black film which contains  $\text{Fe}_3\text{O}_4$ .

**Question by Julius J. Harwood, Office of Naval Research, Navy Dept., Washington, D. C.:**

Did any of the results indicate the reasons why iron enhances the corrosion resistance of the Cu-Ni alloys?

**Authors' Reply:**

With regard to the question raised by Mr. Harwood, we would like to know more about the mechanism by which iron improved the erosion resistance of cupro nickel alloys. We have many reasons to believe that it functions by speeding up the rate of formation and decreasing the permeability of corrosion product films. This is shown by weight loss vs. time curves which show a greater decrease in rate of weight loss with time in the case of the alloys that contain iron as compared with those that do not. Furthermore, it is known that the presence of iron compounds in the water, coming from other sources, will be helpful in increasing the protective effect of the corrosion products that form on cupro nickel alloys.

The acquisition of knowledge as to the exact nature of the protective films and the role of iron in their behavior has been hampered by the difficulty of preserving the films without change when they are removed from the environment in which they have formed. This restrains the use of x-ray or electron diffraction techniques which otherwise might be expected to be helpful.





# NACE News

## Atlanta Section Organizes, Applies for Charter

### Philadelphia Panel Discussion Planned For Oct. 10 Meeting

A panel discussion on corrosion will be held by Philadelphia Section at the Poor Richard Club, 1319 Locust St., Philadelphia on October 10. This is the first of four meetings scheduled by the section.

Other meetings planned include:

December 12—Poor Richard Club, "Recent Trends in Stainless Steels and Other High Alloys," by Walter A. Luce, The Duriron Co.

February 13—Poor Richard Club, "Corrosion Inhibitors," by Paul Cardwell, of Dowell, Inc.

May 8—Poor Richard Club. Program to be arranged.

### Southwestern Ohio Set Petroleum Rust Preventives Program

Petroleum rust preventives will be discussed by S. A. Vago, Texas Company sales engineer at the October 28 meeting of Southwestern Ohio Section. The dinner meeting will be held beginning at 6:30 p.m. at Shullers Restaurant, Reading, Ohio. This is the first of six scheduled meetings of the section.

Other meetings of the section have been planned as follows, all to be held beginning at 6:30 p.m. at Shuller's Restaurant, Reading, Ohio except as noted:

November 25—The Corrosion Resistant Properties of Copper and Its Alloys by A. W. Tracy, American Brass Co., Waterbury, Conn.

January 27—International Nickel Co. film "Corrosion in Action." Discussion by Roy McDuffie, professor of metallurgy, University of Cincinnati.

March 31—An address by Charles G. Munger, technical director, Amercoat Corp., South Gate, Cal.

April 28—Trip through Middletown Division mill of Armco Steel Corp., W. F. Johnson, training supervisor in charge. Dinner meeting, cocktail lounge, Manchester Hotel, Middletown, Ohio.

May 26—Activities at Fernald by Wm. E. Shaw, department head, National Lead Company of Ohio.

The 1953 Conference and Exhibition of the National Association of Corrosion Engineers will be held March 16-20 with headquarters at the Sherman Hotel, Chicago.



### NACE MEETINGS CALENDAR

Oct.

6—North Texas Section. Dallas.

21—Chicago Section. Chicago Engineer's Club. Vinyl Resins and Rigid Polyvinyl Chloride by Harry M. Zimmerman, B. F. Goodrich Co.

28—Southwestern Ohio. Shuller's Restaurant, Reading, Ohio. Petroleum Rust Preventives by S. A. Vago, Texas Company.

### Seven Chicago Section Meetings Are Arranged

All seven of the planned meetings of Chicago Section beginning with the September 16 meeting will be held at Chicago Engineers Club, 314 South Federal St. The September meeting, first of the 1952 fall season, included a showing of the International Nickel Company film, "Corrosion in Action."

Other meetings have been planned for October 21, November 18, January 20, February 17, April 21 and May 19.

### Monthly Meetings Are Set by North Texas

Meetings of North Texas Section have been scheduled for first Mondays monthly beginning at 6:30 p.m., the October 6 meeting being planned for El Phenix restaurant, Dallas.

At the September 9 meeting, held at Grand Prairie, Texas, when the International Nickel Co., Inc., film was shown there were present 43 members and 30 guests.

### Hugo W. Wahlquist Now Power Correspondent

Hugo W. Wahlquist, with Ebasco Services, New York City has accepted the position of Corrosion Correspondent for the Power Industry. He will advise the editor of the magazine on matters pertaining to the power industry when called on and supply copy annually for a Topic of the Month in the Power field. He succeeds Irwin C. Dietze, Department of Water and Power, City of Los Angeles.

### Will Become First South East Region Separate Section

A request has been made for a Section Charter for the Atlanta, Georgia Section of the National Association of Corrosion Engineers. This is the first separate section organized in the South East NACE Region, and will be the twenty-sixth local organization in the association.

Eighteen NACE members, at a luncheon in Atlanta June 13, formally organized, voted to request a charter and elected officers as follows:

J. Flynn Johnston, American Telephone and Telegraph Co., Atlanta, chairman; Raymond F. Trapp, Midwestern Engine & Equipment Co., Hapeville, Ga., vice-chairman; Alex M. Erganian, Pipe Line Service Corp., Atlanta, secretary-treasurer.

### Westervelt Is Elected

John P. Westervelt, Pontiac Refining Co., Corpus Christi, Texas is now secretary-treasurer of Corpus Christi Section. Mr. Westervelt succeeds Maurice N. Emery, resigned.

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# All But Three Chairmen Designated for Chicago Five Papers for Protective Coatings Symposium Are Listed by Whiteneck

## Centrifugal Scrubber's Removal of Dust From Pipe Lines Explained

Elimination of dust in natural gas pipe lines is the subject of an article by P. F. Best published in "The Project Engineer," June, 1952. Although there are in general three methods of extracting dust from natural gas lines only the dry or centrifugal type scrubber is described in the article.

Dust in natural gas pipe lines travels with the gas from fields in the Southwest through the long transmission lines, into the distributing lines of cities in the North and East and finally right into the home and factory appliances and machines of consumers.

### Abrasion Wears Equipment

In booster compressor stations, dust abrasion causes rapid wear of cylinder walls, piston rings and packing. It erodes valves and to some extent, causes wear in sharp bends of pipe lines. In consumers' equipment, dust abrasion also occurs and sometimes dust particles plug orifices.

In the dry type of scrubber, the dust-laden gas passes through a parallel arrangement of small, high efficiency vortex collectors with the dust being collected in the bottom of the scrubber and the clean gas passing through the outlet at the top. Most of the dust particles in natural gas pipe lines are comparatively large in size, about 60 microns. It is claimed that the dry scrubber is highly efficient in removing these large particles.

The author cites three samples of pipe line dust which were taken from the gas stream of natural gas pipe lines. Particle size analysis of the three samples are given in table form. Particles of each sample pile were separated into 7 groups, the largest group composed of particles of 60 microns and over and the smallest group of particles measuring 10 microns or less. The sample containing the finest dust had three percent of particles 10 microns or less. The coarsest sample had one-tenth of one percent particles of 10 microns or less. In all three samples, most of the particles were 60 microns or greater.

### Efficient on Coarse Particles

Although the dry scrubber is less efficient in removing fine than coarse particles of dust from lines, it is claimed it is 99.12 percent efficient in removing the finest dust from the gas stream. Efficiency increases to 99.4 percent on coarse particles of dust.

Efficiency of the scrubber increases with the volume and velocity of the gas stream despite the corresponding higher charge of dust. Capacity of the scrubber is limited only to the working pressure of the working parts, about 20 PSI. The author points out that it is impossible to overload the scrubber.

Five papers to be presented during the Protective Coatings Symposium at Chicago, March 16-20, 1953 have been designated. L. L. Whiteneck, chairman of the symposium listed the papers as follows:

Coating Research and Application in Atomic Energy Operations by C. D. Watson.

Investigation of Protective Coatings for High Temperature Underground Pipelines by C. R. Stauffer.

Nature and Theory of Filiform Corrosion by M. VanLoo.

Effects of Composition of Steel on the Performance of Organic Coatings in Atmospheric Exposure by F. L. LaQue and J. A. Boylan.

Weld Seam and Weld Flux Effects on Coating Life by A. J. Liebman.

## L. D. Cook Certified Chairman at Detroit

L. D. Cook, Wyandotte Chemical Corp., Wyandotte, Mich. has been certified as chairman of Detroit Section. Other officers of the section elected and serving as of July 1 are: W. R. Cavanagh, Parker Rust Proof Co., Detroit, vice-chairman; P. G. Coates, Michigan Bell Telephone Co., Detroit, treasurer and D. F. Finlay, Hinchman Corp., Detroit, treasurer.

## Reprints Will be Made Of Stress Corrosion Symposium Papers

Reprints of the "Symposium on Sulfide Stress Corrosion," consisting of papers 1 through 4 presented at the NACE Galveston meeting in March, plus an introductory paper reprinted from the Canadian Mining and Metallurgical Review will be reprinted from the October issue of Corrosion. NACE is providing a number of copies for resale to those interested in this problem.

Single copies will be sold for \$1 each; or 50c each for five or more copies delivered to one address. Those interested may inspect the symposium in this issue's Technical Section, of which it is the major part.

## Canadians Seek Region

Membership of Canadians in the National Association of Corrosion Engineers has more than doubled in five months, according to Organizational Bulletin No. 2 issued by G. I. Russell from The Gas Building, Chatham, Ontario. Canadian members of the association propose to ask the NACE to establish a Canadian Region in October.

More than 4190 copies of Corrosion are being mailed monthly.

## Second Co-Chairmen Prepare for 1954 Kansas City Meeting

All but three chairmen of the principal subdivisions of the 1953 Chicago Conference Technical Program have been named. They are: Corrosion Principles and Pipe Line Symposia and the Pipe Line Corrosion Round Table. In the case of the pipe line items naming of chairmen has been delayed pending an expression of the wishes of the pipe liners.

In some instances a second co-chairman has been named. This co-chairman has agreed to act as chairman of a symposium in the Tenth Annual Conference to be held at Kansas City in March, 1954. Participation in 1953 technical program arrangements is expected to provide valuable experience.

### Chairmen Named

Chairmen and co-chairmen named for the various subdivisions of the technical program so far by Technical Program Chairman H. W. Schmidt, Dow Chemical Company are as follows:

Cathodic Protection—E. P. Doremus, Cathodic Protection Service, Houston, chairman.

Chemical Industry — C. A. Coberly, Mallinckrodt Chemical Works, St. Louis, chairman. E. G. Holmberg, Alloy Steel Products Co., Linden, N. J., and G. F. Lockeman, Procter & Gamble Co., Cincinnati, co-chairmen.

Corrosion Principles—J. V. Petrocelli, Patent Button Company, Waterbury, Conn.

Educational Lectures—R. B. Hoxeng, U. S. Steel Co., Pittsburgh, chairman. R. C. Weast, Case Institute, Cleveland, co-chairman.

Electrical and Communication—C. H. Fellows, Detroit Edison Co., Detroit, chairman. W. D. Sanderson, Detroit Edison Co., Detroit and R. M. Lawall, American Telephone and Telegraph Co., Cleveland, co-chairmen.

High Temperature—J. B. Rutherford, Babcock & Wilcox Tube Co., Beaver Falls, Pa., chairman. F. A. Fritzen, Haynes Stellite Co., Kokomo, Ind., co-chairman.

Oil and Gas—H. L. Bilhartz, Atlantic Refining Co., Dallas, chairman. R. C. Buchan, Humble Oil & Ref. Co., Houston, co-chairman.

Protective Coatings—L. L. Whiteneck, 1333 El Embarcadero, Long Beach, Cal., chairman. N. K. Senatoroff, Southern Counties Gas Co. of Cal., Los Angeles, co-chairman.

Refinery—R. S. Treseder, Shell Development Co., Emeryville, Cal., chairman.

Water—V. V. Kendall, National Tube Co., Pittsburgh, chairman. S. T. Powell, 330 N. Charles St., Baltimore, co-chairman.

General Corrosion Round Table—C. F. Pogacar, Atlantic Refining Co., Philadelphia, chairman. L. E. Stout, Washington University, St. Louis, co-chairman.

## TP-8 Sub-Group Outlines Plans to Probe Gulf Coast Cooling Water Problems

A proposed sub-group of Technical Practices Committee 8 on Corrosion by Waters is suggested by Charles P. Dillon of Carbide and Carbon Chemical Co., Texas City, Texas to organize and develop a cooperative investigation of problems pertaining to corrosion resulting from the use of fresh, brackish and salt waters in cooling towers, especially of process equipment on the Texas-Louisiana Gulf Coast. Organization of this committee was discussed and approved following a meeting of interested engineers at the Galveston Conference in March. V. V. Kendall, chairman of TP-8 authorized formation of the committee.

Proposed subjects for investigation by Technical Practices Committee 8-A have been outlined and a selected group of engineers in Gulf Coast plants has been asked to express views respecting the work the committee should undertake.

Mr. Dillon points out numerous investigations are now being conducted into problems associated with cooling waters and that the committee would be of material service in coordinating the investigations, eliminating duplicated work and making discoveries available to all concerned.

## Inhibited Hydrochloric Acid Corrosion Is Discussed at Houston

Paul H. Cardwell, chief chemist of Dowell Incorporated, Tulsa, Okla. addressed Houston Section's September 9 dinner meeting on "The Microstructure and Corrodibility of Steel in Inhibited Hydrochloric Acid Solutions." Dr. Cardwell presented evidence that poorly resolvable pearlitic structures, local grain coarsening and Widmanstatten structures increase corrodibility, whereas a structure with well developed pearlite in a steel with ASTM ferritic grain size 7 or 8 is resistant. It was brought out that the effectiveness of inhibitors is enhanced by wetting agents, and while reasons for this are not understood, it is assumed they may be associated with contact angles of hydrogen bubbles on the steel surface.

M. E. Holmberg, Houston consulting metallurgist, reviewed some of the findings of Phillips Petroleum Company which developed further the theme microstructure is important in some services.

James Woodburn, head of Rice Institute's mechanical engineering department, gave a talk at dinner on the interrelationship among industry, universities and engineering students and their mutual responsibilities.

NACE sells subscriptions on a calendar year basis of McBee punch cards on which are printed abstracts of corrosion literature published in more than 500 periodicals at home and abroad. More than 2000 cards are issued annually. They are pre-indexed according to the punch card index system by topics, periodical in which published, first and second authors, and year.

## NACE's '54 National Meeting Place Set

The 1954 Conference and Exhibition of the National Association of Corrosion Engineers will be held March 15-19 inclusive at Kansas City. The exhibition will be held at the Kansas City Municipal Auditorium. There was a meeting of the National Association of Corrosion Engineers at Kansas City in 1946.

The name of the headquarters hotel has not been revealed.

## Washington University Registers Over 100 at Corrosion Short Course

More than the stipulated 100 registrants, from places as distant as Colombia, South America and Calgary, Canada, registered for the Washington University, St. Louis Short Course on Corrosion. The course was held September 8-12. Registrants from Maine, West Virginia, Louisiana, Los Angeles and New York City, among other places were present.

Of those attending about 80 were from the vicinity of St. Louis. A tour of the Anheuser-Busch plant was attended by some registrants.

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## University of California, Berkeley Plans Corrosion Short Course for February 2-6

### Shreveport Section's Third Corrosion Control Short Course Is Held

The Third Annual Corrosion Control Short Course sponsored by Shreveport Section was held at Centenary College, Shreveport, September 18-19.

The program included:

September 18—Registration. Introduction by Tom L. Holcombe. "Corrosion in Action," motion picture produced by the International Nickel Co., Inc.

Fundamental Corrosion Theories, E. P. Doremus. Knotty Problems round table discussion.

Cathodic Protection Design and Installation, A. W. Peabody.

Protective Coatings, Robert L. Bullock.

Fellowship hour and banquet.

September 19—Instruments for Field Testing and Procedures, M. E. Parker.

Better Results Through Improved Operation, M. E. Parker.

Improving Field Maintenance, Methods, Suggestions, Information and Data Which Should be Furnished Central Office, C. L. Woody.

Evaluating Internal Corrosion, B. H. Pickard.

Insulating Well Flow Lines, Lynn H. McGuffy.

Mitigation of Tubing and Casing Corrosion, C. P. Siess.

### Electrochemical Society Plans 2-Day Corrosion Symposium at Montreal

A two-day symposium on corrosion will be a feature of the Montreal Meeting of The Electrochemical Society, October 26-30. A full-day session on high temperature corrosion of metals will be held Monday, October 27 while Tuesday morning will be devoted to a general session on corrosion and Tuesday afternoon to a session on passivity and inhibitors. Other scheduled sessions are on electrodeposition, industrial electrolytic, electric insulation, battery and electrothermics.

Several plant trips, including an all-day visit at the Aluminum Company of Canada's plant at Arvida, will be held. Also planned are trips to McGill University's electronics plant, Canadian Copper Refiners and Noranda Copper and Brass, Ltd.

### Lowe Will Direct Steel Founders' Program

James H. Lowe has been named product development director of Steel Founders' Society of America. Formerly technical advisor and supervisor of E. I. duPont de Nemours & Co.'s military explosives division, Mr. Lowe also was technical and research director of Wehr Steel Company, Milwaukee.

A five-day Corrosion Short Course has been scheduled for February 2-6 by University of California, Berkeley, with the National Association of Corrosion Engineers as co-sponsor. Full details of the course have not been determined but the following schedule of topics and times is contemplated:

#### Monday, Feb. 2—Fundamentals

8-9 am—Registration.  
9:9:15 am—Opening of meeting.  
9:15-10:15 am—Introduction to study of corrosion. Definitions, magnitude, types, sources of information.  
10:25 am to 12 m—Fundamentals of corrosion. Physicochemical factors. Metallurgical and mechanical factors.  
1:30-3:20 pm—Fundamentals of corrosion continued.  
3:30-5 pm—Stress corrosion.

#### Tuesday, Feb. 3—Materials of Construction

9-10:50 am—Ferrous metals.  
11-12 m—Non-ferrous metals.  
3-5 pm—Special metals and alloys.

#### Wednesday, Feb. 4—Coatings

9-10:50 am—Chemical conversion coatings.  
11-12 m—Organic protective coatings.  
1:30-2:30 pm—Organic coatings continued.  
2:40-5 pm—Metal coatings.

#### Thursday, Feb. 5—Environment and Its Treatment

9-11 am—Process industry.  
11:10-12 m—Public water supplies.

#### Cathodic Protection

1:30-3 pm—Cathodic protection principles.  
3:10-5 pm—Cathodic protection practices.

#### Friday, Feb. 6—Special Topics

9-10:30 am—Corrosion testing and evaluation.  
10:40-12 m—Design of equipment from corrosion standpoint.  
2:40-2:50 pm—Metals at high temperatures—Oxidation and structural stability.  
4-5 pm—Role of the Corrosion Engineer in Industry.  
(Summary to close the course.)

### Heavy Registration Is Forecast at New Orleans

Heavy early registration for the 1952 South Central Region meeting at New Orleans indicates the attendance record will be broken again. Committeemen working under Clifford L. Barr, General Arrangements Committee chairman and John E. Loeffler, Technical Program chairman, have prepared a program geared to the interests of the region's membership.

Early publicity provided by the New Orleans group made it possible to get information about the meeting into the hands of many non-members of NACE.

## Corrosion Problems

E. A. TICE, Editor

Corrosion Engineering Section, The International Nickel Co., Inc., 67 Wall St., New York 5, N. Y.

Submit questions and answers for this column in duplicate to the editor. All questions become property of NACE. Questions and Answers may or may not be published under this heading, and may at discretion, be answered by mail directly. Answers to questions are solicited. Authors of questions will remain anonymous to readers while authors of answers may remain anonymous if they request it.

### QUESTION

No. 94—(From one of our French associates.) This problem is in reference to petroleum refining. Gasoline vapors, after leaving the top of the distillation towers, generally receive an ammonia injection to neutralize the free hydrochloric acid resulting from hydrolysis of  $MgCl_2$  and  $CaCl_2$  contained in the crude. This ammonia gas injection is generally regulated so that the water pH, after condensing mixed gasoline and water is as close to 7 as possible.

In spite of this injection, we have observed that in the temperature range of 100 degrees C or somewhat higher, high corrosion rates for steel equipment prevail. Similarly, gasolines already neutralized with ammonia and insufficiently decanted after condensing, do sometimes, when reheated for rerunning show again the same corrosive properties in the above mentioned temperature range.

We should like to know whether other refineries using ammonia as a neutralizing agent have observed the same effect, and whether any research has been conducted to determine the efficiency of this process at various temperatures. Literature references connected with this problem would be most welcome.

### ANSWER

Mr. Laurent cites a case in which severe corrosion of steel equipment was observed in a condensing system or parts thereof operating at temperatures in the range of 100 degrees C and into which ammonia was being injected to neutralize free hydrochloric acid. In the temperature range cited, aqueous solutions of ammonium chloride, the product of the neutralization reaction, become quite corrosive. Experiments conducted at the Research and Development Laboratories of The Atlantic Refining Company at Philadelphia, Pa., indicated that the corrosion rates of mild steel in an 0.1 N ammonium chloride solution increased rapidly with temperature and were in the order of 0.006, 0.050 and 0.300 inches per year at 25, 65 and 100 degrees C, respectively. Other experiments also indicated that at temperatures in the range of 100 degrees C the corrosion rates of steel in 0.1 N hydrochloric acid solutions adjusted to pH levels of 4, 6 and 8 with 0.1 N ammonia hydroxide were greater than 0.100 inches per year.

This temperature effect on the corrosion of steel in aqueous ammonium chloride has in part made it necessary in the past to use an alloy, such as Monel, cupro-nickel, admiralty, etc., which is resistant to the medium under conditions existing in condensing equipment, as

a protective liner or substitute for steel equipment. At the present time, there is considerable development in progress on the application of filming type organic inhibitors to control condensate water corrosion. These organic inhibitors are used in conjunction with or in place of ammonia injection. In either case, there are indications that these inhibitors are more effective than ammonia neutralization alone and that the necessity for using alloys to overcome the shortcomings of ammonia control may be obviated.

The residual condensate water in a poorly decanted gasoline although exhibiting a neutral or alkaline pH at 25 degrees C will behave as described above upon reheating. It appears that the best recommendation in Mr. Laurent's case would be to take steps to improve water separation and thereby reduce the carry over of aqueous ammonium chloride with the gasoline. Organic inhibitors may also be an answer to the problem providing the reheating is carried

out in a tubular exchanger in which the temperature of the hot fluid does not exceed 230 degrees C. However, if reheating is accomplished in a direct fired heater, organic inhibitors are usually ineffective since the skin temperatures of the tubes generally exceed the decomposition temperatures (200-250 degrees C) of the inhibitors.

A review of the literature indicates there has been little research on Mr. Laurent's problem per se. Generally, the problems in using ammonia neutralization to control hydrochloric acid corrosion have been complicated by the fact that hydrogen sulfide is also present. Therefore, the majority of the works published have considered systems containing both corrosive agents. These works, however, would be of interest to Mr. Laurent. Rather than list an extensive bibliography at this time, a few of the more recent publications which have excellent attending bibliographies are given below.

(Continued on Page 6)

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## Corrosion Problems—

(Continued From Page 5)

### Bibliography

1. Arthur Marks. Corrosion Protection in Refineries. *Proc. API*, 17 (III) 1936.
2. L. L. Davis et al. Laboratory Control of the Corrosion of Distillation Equipment and Desalting Processes. *Proc. API*, 19M (III) 1938.
3. E. Q. Camp. Corrosion Prevention in Processing Sour Crudes. *Corrosion*, 4, 371-398 (1948).

Listed below are a few of the latest articles concerning the use of organic inhibitors which may prove a partial answer to Mr. Laurent's problem:

1. M. J. Wilcox et al. How to Control Internal Corrosion of Crude Still Condensers. *Oil & Gas J.*, 50, No. 45, 290-293, (1952) Mar. 17.

2. F. E. Love. Production-Proved Inhibitor Gets Results in Refineries and Natural Gas Plants. *Ibid.*, 310-312.

By: William A. Hall, The Atlantic Refining Company, Philadelphia.

No. 102—What economy might be obtained and what potential gradient expected, by using an abandoned bare steel pipe line, parallel and 30 feet away, and some 5000 feet long, as a ground bed for rectifier for a new coated pipe line in an area affected slightly by stray currents?

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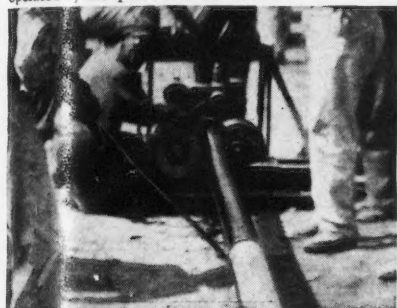
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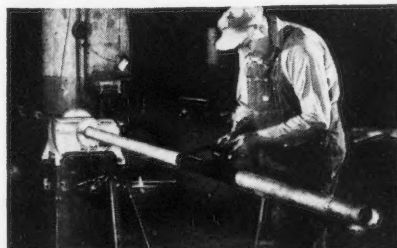
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WHITE, ROBERT U., Standard Pipe Line Co., Box 752 Bakersfield, Cal.  
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HAGERMAN, JOHN R., Barrett Div., Allied

Chem. & Dye Corp., 205 W. Wacker Dr., Chicago 6, Ill.

POOLE, JAMES E., Keystone Asphalt Products Co., 101 East Ontario, Chicago, Ill.

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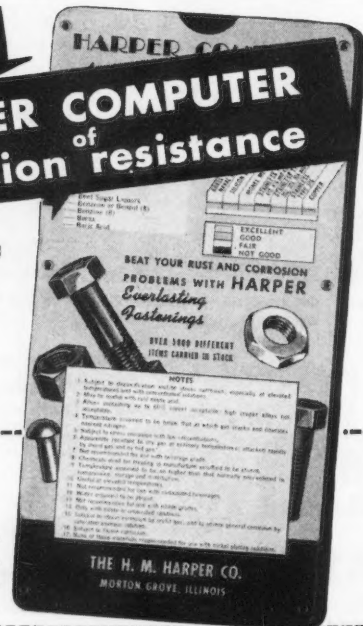
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# TECHNICAL TOPICS

## MACHINING 18-8 STAINLESS STEEL—

### Metallurgical Considerations

Norman S. Mott  
Chief Chemist and Metallurgist

Most of the headaches associated with the machining of stainless alloys can be cured with machining experience, but some of them are associated with metallurgical considerations. This applies both to the wrought and the cast materials. Too often the underlying causes and the possibilities for their correction are not well understood. The purpose of this discussion is to review these causes, and to indicate the steps which may be taken to eliminate them.

The basic difficulties experienced in machining of stainless alloys are:

1. The metal is too hard . . . The cutting tool cannot penetrate or too much tool pressure is required.
2. The metal is too tough . . . It tears away instead of breaking up into chips.
3. Frictional or galling characteristics are excessive . . . Chips adhere to the tool tip, resulting in the balling up of the cut metal.
4. Microstructural non-uniformity or segregation . . . This causes hard spots and results in rough or uneven machined surfaces.
5. The metal work hardens . . . This results in a blunt tool and a polishing rather than a cutting action. Let us review each of these difficulties in turn:

**Hardness:** Hardness in 18-8 stainless, such as to cause difficult machining, is not an inherent characteristic. It is usually found in the form of work hardening, either from cold rolling or from hot working to too low a temperature. Although machining op-

erations have been conducted up to as high as 350 Brinell, this requires a slow special technique and for practical purposes such hard metal should be soft annealed by water quenching from 1950-2100°F.

**Toughness:** Correctly heat treated 18-8 stainless in the condition for maximum corrosion resistance is very tough and ductile. These are desirable characteristics from a mechanical viewpoint, but they are pretty tough on machinability. Experience has shown that machining difficulties can be minimized through the addition of an embrittling agent such as selenium, sulphur or phosphorus. And when properly controlled, maximum machinability with a minimum loss of corrosion resistance can be accomplished.

**Galling:** Galling tendencies, which are associated with softness and ductility, are also largely eliminated by controlled additions of various alloying elements.

**Hard Spots:** Hard spots are caused by microstructural segregations such as carbides and other hard phases. The machining tool in passing over these areas does not cut properly and gives a raised and usually a glazed surface, producing a so-called "orange peel" effect. Correct quench annealing heat treatment is required in order to eliminate such a condition. Another cause of hard spots may be burnt-in pieces of molding sand, a condition sometimes found in poor quality castings.

**Work Hardening:** Work hardening is a universal characteristic of the 18-8 stainless steels. Pressure by the machine tool tends to cold work the surface and make it hard. To offset this, there are a number of machinists' tricks which are somewhat beyond the scope of this discussion. (Further data on the machining techniques can be found in J. J. Roberts' paper, "Don't Fear Threading of Stainless.")

Copies of this article reprinted on heavy stock for convenient filing are available on request.



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## Chromium-Frit Coatings Limit Oxidation Corrosion of Molybdenum

**B**ECAUSE of its high melting point (4750 degrees F), molybdenum offers possibilities for use in aircraft jet engines. If molybdenum is to be used at jet-engine temperatures, however, it must be protected from rapid oxidation.

The National Bureau of Standards has recently conducted a study of protective coatings for molybdenum composed of chromium and frit (glass). Results indicate that such coatings greatly extend the useful life of molybdenum at high temperatures, giving better protection than either chromium or ceramic coatings alone. Various chromium-frit coatings were bonded to molybdenum specimens, then subjected to oxidation tests under tension in the range of 1500 to 1800 degrees F and to flame tests in the range 2000 to 3000 degrees F. At temperatures of 1500 to 1800 degrees F the coated specimens lasted for 1000 to 3000 hours. At 2800 degrees F, with no applied load, protection for as much as seven hours was attained, enough to be valuable for some applications. The investigation was conducted by D. G. Moore and associates of the NBS enameled metals laboratory, under the sponsorship of the National Advisory Committee for Aeronautics.\*

The present NBS study is one phase of a continuing program for development of ceramic protective coatings for metals and alloys. This NACA-sponsored program has resulted in several successful NBS coating formulations

\* This NBS study is reported in detail in "Study of Chromium-Frit-Type Coatings for High-Temperature Protection of Molybdenum", by D. G. Moore, L. H. Bolz, J. W. Pitts and W. N. Harrison, NACA Technical Note 2422 (July, 1951).

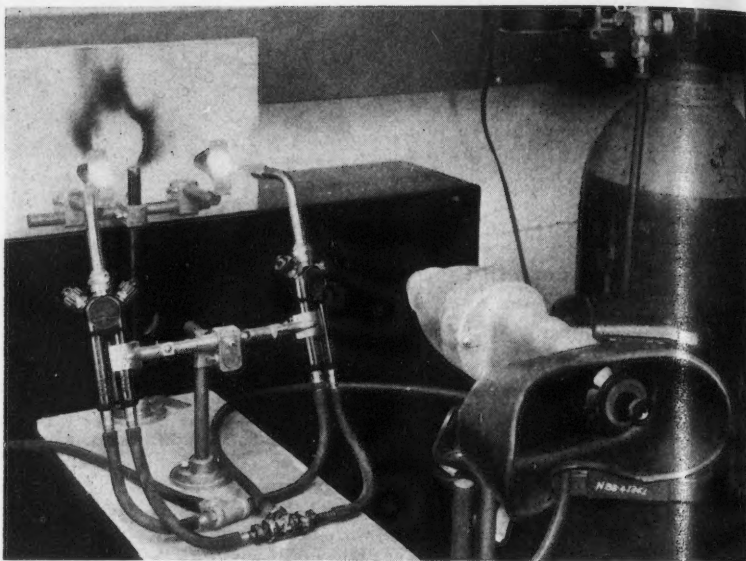


Figure 1—Apparatus for flame-testing coated molybdenum specimens at the National Bureau of Standards. Specimen surface temperatures, which ranged from 2000 to 3000 degrees F, were observed by means of an optical pyrometer (right of center). Chromium-frit coatings were found to add substantially to the life of the molybdenum.

that are now in regular commercial production.

In this new study, molybdenum specimens were first coated with one of several powdered chromium-bearing base coats, which in some cases also contained some frit (glass). After firing of the base coat, in either a hydrogen or an argon

atmosphere, a ceramic seal-coat containing no chromium was applied to some of the specimens. Test results indicate that the most durable coatings resulted from application of a glass-free chromium base coat, followed by a glass seal coat. Ad-

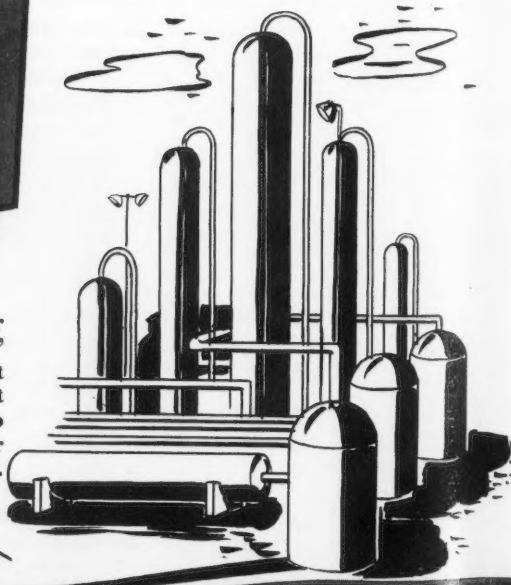
(Continued on Page 11)

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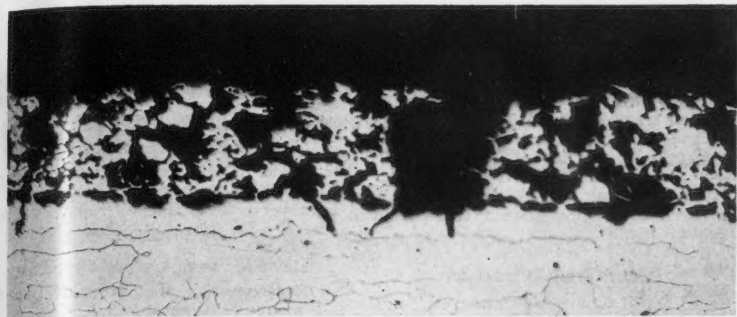


Figure 2—Photomicrograph of molybdenum specimen coated with glass seal-coat M-13 on top of chromium-frit coating M-39. The specimen was subjected to thermal shock for 25 cycles from 1800 degrees F prior to sectioning. The glass seal-coat apparently tended to seal thermal-shock cracks that developed in the base coat. Top (dark grey) layer: plastic mount. Second (narrow black) layer: glass seal-coat. Third (variegated grey-and-white) layer: chromium-frit coating. Fourth (narrow white) layer: diffusion layer. Bottom (near-white) layer: molybdenum. (X150, etched in nitric acid.)

## Chromium and Frit—

(Continued from Page 10)

dition of a third coat, consisting of lower-melting glass, resulted in a coating showing the highest durability of all those investigated.

Microscopic examination of sections shows that the chromium of the base coat has a somewhat porous structure, formed by the diffusion-welding of the chromium particles to each other and to the molybdenum base. The glass is subsequently absorbed into this structure during the firing of the seal coat. Although thermal strain and rapid creep tend to cause cracking of the coatings, adherence is excellent and the glassy layer tends to reseal the fissures, so that cracking does not result in rapid failure.

## BOOK REVIEWS

**Lead in Modern Industry.** The Lead Industries Association, 420 Lexington Ave., New York, N. Y. 230 pages, 6 x 9 inches, cloth. Per copy, \$1.50.

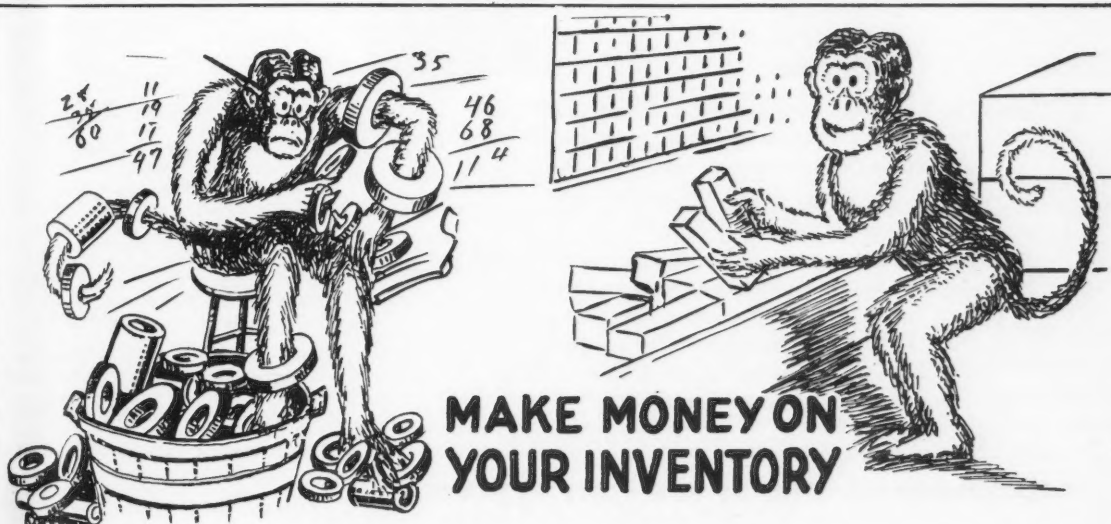
While the book is written in layman's terms, it also includes technical data and methods which are useful to the engineer. There is a short history of lead from 3000 BC specimens to the present. A description of processes and mining from the raw ore to the finished product or end use is given. The concentration of lead ore, mostly in the form of lead sulfide and smelting the lead ore concentrates and the refining

process are explained. The most widely used refining process according to the book is the Parkes process.

Secondary lead and world consumption of lead is discussed. A chapter is devoted to the importance of lead in modern living. Lead is used in fertilizer. It is used by scientists experimenting and working with X-rays and gamma rays. Artists use lead in making such things as beautiful glass art objects.

For the technically minded, the book delves into the fabrication, extrusion, rolling and various types of castings of lead. Three chapters devote considerable space to the use of lead in storage batteries, cable covers and modern chemical construction. Corrosion resistance of lead and lead alloys, corrosion rate interpretation, fatigue and stress corrosion, galvanic corrosion and like subjects are discussed. The uses of lead in soldering and welding are explained and the composition of commercial pig lead is tabulated.

Several chapters are devoted to the use of lead in protective coatings such as metallic coatings, lead pigmented paints and other chemical compounds for industrial use. Lead for ammunition both in war and peace time is discussed and the use of lead tetraethyl in gasoline is listed as are some of the more important uses of the metal. Considerable space is devoted to the safe handling of lead and its products and many charts and data tables concerning the specifications and properties both physical and chemical for both lead and lead alloys are stated. The book is well illustrated.



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## Metal Keeps the Peace Is Theme of Congress

The 34th National Metal Congress and Exposition, sponsored by the American Society for Metals is scheduled for Oct. 18-24 in Philadelphia, Pa. Theme of the meeting is "Metal Keeps the Peace."

Among the many papers for presentation were:

### High Temperature Phases

Microconstituents in High Temperature Alloys.

Sigma Formation and Its Effects on the Impact Properties of Iron-Nickel.

Mechanism of the Carburization of Some Stainless Steels.

The Electrolytic Separation and Some Properties of Austenite and Sigma in

18-8-3-1 Chromium-Nickel-Molybdenum-Titanium Steel.

### Creep Rupture and Recrystallization

Creep-Rupture and Recrystallization of Monel from 700-1700 degrees F.

Influence of Grain Size on High Temperature Properties of Monel.

Creep and Rupture of Chromium-Nickel Austenitic Stainless Steels.

Recrystallization and Grain Growth in Alpha Brass.

### Phase Transformation

The Effect of Composition on the Temperature of Spontaneous Transformation of Austenite to Martensite in 18-8 Type Stainless Steel.

The Effect of Silicon on the Tempering of Martensite.

### Mechanical Properties

X-Ray Measurement of Residual Stress in Hardened High Carbon Steel.

Plastic Stress-Strain Relations of Alcoa 14S-T6 for Variable Biaxial Stress Ratios.

### Temper Brittleness

The Effect of Various Heat Treating Cycles Upon Temper Brittleness.

Transverse Mechanical Properties in an SAE 1045 Forging Steel.

### Elevated Temperature Properties

Some Properties of a Nodular Iron at Elevated Temperatures.

Accelerated Strain Aging of Commercial Sheet Steels.

### Seminar

Crystal Growth and Crystal Boundary Techniques.

Deformation of Single Crystals, Radioactive Tracers.

### Behavior of Metals at Low Temperatures

Behavior of Single Crystals and Pure Metals.

Influence of Mechanical Variables, Influence of Metallurgical Factors.

### Gases in Metals

Gases in Nonferrous Metals and Alloys.

### Metallurgical Tools for Alloy Conservation and Increased Production

Significance of Chemical Analyses in Alloy Classification and Mechanical Properties.

Progress in Spectrography and X-Ray Diffraction Analyses and Techniques.

### Welding

Chromium-Recovery During Submerged-Arc Welding.

Corrosion of Structural Spot Welds.

Further Studies of the Crack Sensitivity of Aircraft Steels.

Relationship of Welding Technique to Penetration and Dilution.

New Aspects in Surface Alloying in Brazing and Related Techniques.

Relation of Preheating to Low-Temperature Cooling Rate Embrittlement and Microcracking.

Initiation and propagation of Brittle Fracture in Structural Steels.

The Continuous Cooling Transformation of Weld Heat-Affected Zones.

Embrittlement of High Strength Ferritic Welds.

Welding and Forming of Titanium.

Evaluation of Brittle Failure Research.

An Investigation on Peening.

Pipe Welding in the Petroleum Refining Industry.

Prevention of Marine Corrosion by Metallizing Systems.

Failure and Defects Encountered in Welded Ship Construction.

### Thermodynamic and Thermal Properties

Scaling of Lead in Air.

Transformation in Cobalt-Nickel Alloys.

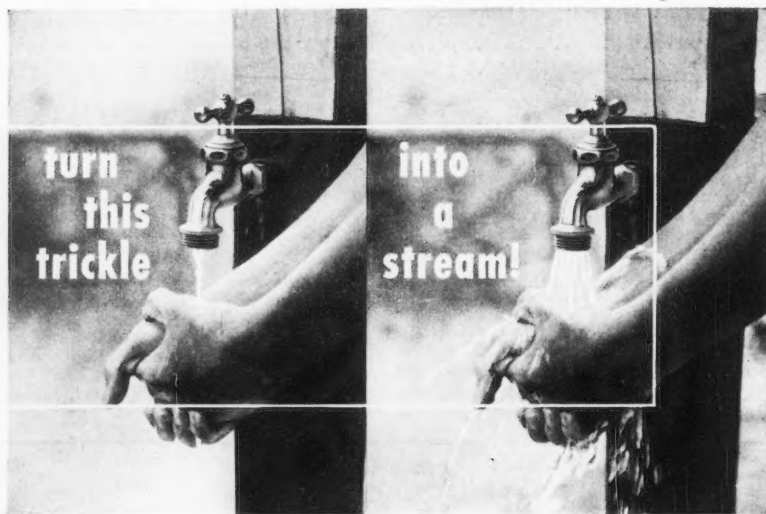
Principles of Zone-Melting.

Some Observations of Sub Grain Formation During Creep in High Purity Aluminum.

Observations of Creep of the Grain Boundary in High Purity Aluminum.

Influence of Chemical Composition on the Rupture Properties at 1200 degrees F. of Wrought Cr-Ni-Co-Fe-Mo-W-Cb Alloys.

High Temperature Oxidation of Some Iron Chromium Alloys.




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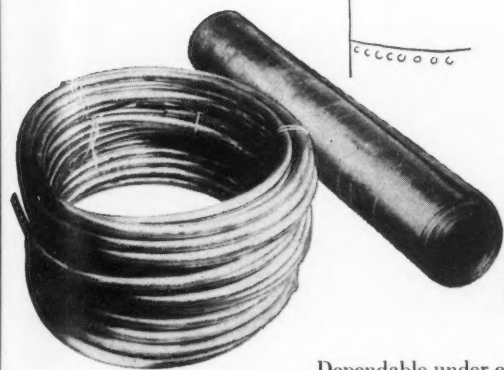
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# NEW PRODUCTS — Materials — Service — Literature

**Barrows Porcelain Enamel Co.,** Cincinnati, Ohio has developed a continuous porcelain enamel fusing furnace for coating pipe. Five different standard 21-foot lengths of pipe, up to two-and-one-half inches OD can be run through the gas fired furnace at the same time. The pipe is used for installations where corrosive and extremely hot liquids and gases are problems.

**The Masonry Resurfacing & Construction Co., Inc.,** Baltimore, Md. announces it has developed a method for underwater repair of wood, steel and concrete piles. Subsurface work is done in the "dry" through the use of a caisson surrounding the pile. By use of the caisson, the pile can be cleaned, burned, welded or painted easily. Since the inspector can see the work, thorough inspection is possible.

**Dressertape,** polyvinyl chloride pipe tape manufactured by Dresser Manufacturing Division of Dresser Industries, Inc., Bradford, Pa., is now being offered in one-inch widths. The new one-inch width is particularly adaptable for wrapping fittings. Ten-mil Dressertape has an insulation resistance of 100,000 megohms and dielectric strength of 10,000 volts. It offers protection against water, salt water, acids, alkalis, oil and soil chemicals.

**Drwal,** a new invisible water repellent for use on exterior stone, formed masonry, brick, concrete and asbestos siding protects these materials against water, dirt and staining according to the Glidden Company, Cleveland, Ohio, maker of the product. Drwal allows masonry to breathe, resists white efflorescence and staining and is unaffected by heat, cold and sunlight. It reduces spalling and disintegration from extreme outdoor weather exposure. Only one coat is required, and water repellency is developed within 24 hours after the application.

**Dow Corning Corp.,** Midland, Mich., has published a "Silicone notebook" describing their Dow Corning 200 fluids. Chemical structure, applications, general properties, boiling and freezing points, volatility and vapor pressure, stability, viscosity-temperature relation, shear resistance, oxidation resistance, heat and flame resistance, water repellency, dielectric properties, chemical behavior, solubility, lubricity, thermal conductivity and other qualities of the liquids are described.

**The National Instrument Co.,** 5005 Queensbury Ave., Baltimore 15, Md., is marketing a pocket measuring microscope with an engraved scale to make linear measurements as small as 0.0005 inch. Among the many uses the microscope has is the inspection of welds, thus eliminating the necessity of sampling analysis, the measuring of diameters of impressions in Brinell tests and inspection of mechanical parts for wear. The microscope is called the "Optometer."

**Minnesota Mining and Manufacturing Co.,** 411 Piquette Ave., Detroit 2, Mich., is producing a new industrial undercoating. The product, EC-1189, is said to form a tough coating, resistant to corrosion, abrasion and weathering. It is said to have excellent sound deadening qualities. Some of the specific corrosion resistant uses are to protect metal buildings in fertilizer, paper pulp, plywood and chemical plants, exteriors of storage tanks, tank trucks and railroad cars. Weathering protection is provided by coating power plant roofs and walls, exteriors of railroad signal bungalows and the bottom exteriors of outdoor switchgear housings. For sound deadening, EC-1189 can be used inside barge deck houses and engine rooms, in the sound trap of air conditioning units and other like uses. Properties include "excellent" adhesion to most materials and surfaces and "excellent" resistance to water, salt water and other corrosive agents. When sprayed or brushed to a one-eighth inch thickness, it dries in about 48 hours. Technical data is available on request from the company.

**Wilkinson Equipment and Supply Corp.,** 6958 S. Wentworth, Chicago, Ill. has developed a new drum spray pump and control unit which allows spraying from the original material container. It is not necessary to transfer lacquers, paints, enamels or special finishes to any special container because the new spray unit fits any standard drum. The new unit positively eliminates surge previously caused by pressure buildup when the spray gun was turned off, the firm maintains. With the new unit, the spray gun is the only control, its action shutting off the pump and spray simultaneously.

**Al-Fin Division** of the Fairchild Engine and Airplane Corp., Farmingdale, L. I., New York has published a 16-page brochure on the "Al-Fin" process of molecularly bonding aluminum and ferrous metals. Briefly, the Al-Fin process consists of the casting of aluminum against an especially prepared surface of the ferrous part to form bi-metallic units having the complimentary physical characteristics of the two metals. According to the company, it is basically a foundry process, and any of the conventional casting methods—sand casting, permanent mold, plaster mold, or die casting can be used.

**The American Hard Rubber Co.,** 93 Worth St., New York, 13, N. Y. is manufacturer of Ace Parian diaphragm valves. The valve body is molded of Ace Parian, a plastic compound in the polyethylene family. The diaphragm may be rubber, neoprene, or polyethylene. The bonnet assembly is sealed off from the solution and the valve has no stuffing box to leak. It can be serviced without removing from the line and all parts are interchangeable. The valves are particularly useful in food processing or in handling corrosive mixtures. Working pressure is 50 lbs. per sq. in. at 77 deg. F.

**The Stonco Electric Products Co.,** 333 Monroe Ave., Kenilworth, N. J. has designed a new illuminating lamp holder to prolong lamp life and provide protection against weather, vibration, pole sway, shock and other conditions contributing to premature lamp failure. The new type of holder uses a silicon rubber cushion-seal that hugs the neck of the lamp in a tight weatherproof seal. The unit is made of non-corrosive cast aluminum with a glazed porcelain heatproof socket.

**Chemaloy,** welding rods for welding aluminum or zinc-base metals can be used for these metals regardless how thin the gauge, down to foil or screening. They will also function with aluminumized or aluminum-coated steel or other base metal and with galvanized surfaces or galvanized iron, the makers say. According to Chemaloy Associates, Gillespie Airport, Santee, Cal., Chemaloy rods eliminate the need for supercleaning, the use of corrosive flux to dissolve surface film or oxides and other specialized equipment.

**Allen D. Stafford, Inc.,** 1814 Richmond Ave., Houston has succeeded Allen D. Stafford as distributor for Amercoat protective coatings on the Gulf Coast. Personnel of the firm includes Edwin O. Stafford and Lucien Miner in the Houston area and Robert A. Steele, Jr. in the Corpus Christi and South Texas area.

**Ball Distributing and Engineering Co.,** Tulsa, Okla., distributor of Gustin Bacon Manufacturing Company's Ultralite glass fiber insulation, has expanded its territory to include a warehouse and sales office at 819 North Olive St., Oklahoma City. Glenn Hughes will be in charge of the Oklahoma City branch. Bill Linihan will be the new office manager.

**The Precision Paper Tube Co.,** 2035 W. Charleston St., Chicago 47, Ill., custom manufactures thread protectors for safeguarding external and internal threads and surfaces on machined parts. The thread protectors are made of kraft fish paper, cellulose acetate or a combination. They can be imprinted with the name and address of the parts manufacturer, part number or other information. The protectors can be crimped or perforated to fit irregularly shaped parts.

**Linatex Corp. of America,** Rockville, Conn. has developed a special form of natural rubber made in Malaya. Linatex is completely resistant to moisture, from three to four times more resilient than other rubbers and is light in weight, having specific gravity of 0.97. Resistance to low temperature is outstanding and Linatex has greater resistance to abrasion, corrosion and vibration than metal or any other type of rubber or plastic makers say. No special training or equipment is needed to install Linatex, the makers report.



# Corrosion Abstracts

## Index To CORROSION ABSTRACTS

Vol. 8      October, 1952      No. 10

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### 1. GENERAL

#### 1.1 Miscellaneous

##### 1.1.1.7.3

A New British Corrosion Organization. T. P. HOAR, Department of Metallurgy, University of Cambridge, Cambridge, England. *Corrosion*, 8, No. 5, 182-184 (1952) May.

The Corrosion Group, a division of the Society of Chemical Industry, was inaugurated in 1951. Objects of the group are to provide a means of communication among chemists, metallurgists, engineers and others concerned with the research and technical aspects of corrosion and to increase the knowledge of corrosion.

Being a part of the Society of Chemical Industry, the Corrosion Group has available for publication of reports of lectures and discussions and other news, the weekly "Chemistry and Industry." Original papers are published in the monthly "Journal of Applied Chemistry."

The Corrosion Group, modeled after the National Association of Corrosion Engineers, is the result of a long-felt need for a means of discussion of corrosion matters in the widest sense. While corrosion problems form only a small part of the activities in the discussions and publications of British technical societies the Corrosion Group is devoted solely to corrosion and all phases of corrosion are discussed under its aegis.

Idea for the Corrosion Group came originally from Dr. R. B. Mears when he was president of NACE. Dr. Mears suggested to Dr. W. H. J. Vernon of the Chemical Research Laboratory, Teddington and to Dr. U. R. Evans of Cambridge University that an organization

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NACE will NOT accept orders for photoprint or microfilm copies of material not published by the association.

### CODE OF AGENCIES SUPPLYING CORROSION ABSTRACTS

AER—Aeronautical Engineering Review, Institute of Aeronautical Sciences, Inc. 2 East 64th St., New York 21, N. Y.

ALL—The Abstract Bulletin, Aluminum Laboratories, Ltd. P. O. Box 84, Kingston, Ontario, Canada.

AWWA—Journal, American Water Works Association, Amer. Water Works Assoc., 521 Fifth Ave., New York 17, N. Y.

BL—Current Technical Literature, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

BLR—Battelle Library Review, Battelle Memorial Institute Library, 505 King Ave., Columbus, Ohio.

BNF—Bulletin; British Non-Ferrous Metals Research Association, 81-91 Euston St., London NW 1, England.

CALCO—Calco Chemical Division, American Cyanamid Corp. Bound Brook, New Jersey.

CE—Chemical Engineering, McGraw-Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

CEC—Consolidated Edison Co. of New York, Inc. 4 Irving Place, New York 3, New York.

EL—Electroplating, 83/85 Udney Park Road, Teddington, Middlesex, England.

EW—Electrical World, McGraw-Hill Publishing Co. 330 W. 42nd St., New York 18, N. Y.

GPC—General Petroleum Corp. of California, 2525 East 37th St., Los Angeles 11, Calif.

INCO—The International Nickel Co., Inc. 67 Wall Street, New York 5, New York.

IP—Institute of Petroleum, 26 Portland Place, London W 1, England.

MA—Metallurgical Abstracts, Institute of Metals, London, England, 4 Grosvenor Gardens, London SW 1, England.

MI—Metallurgia Italiana. Associazione Italiana di Metallurgia. Via S. Paola, 10, Milano, Italia.

MR—Metals Review, American Society of Metals, 7301 Euclid Ave., Cleveland 3, Ohio.

NALCO—National Aluminate Corp. 6216 West 66th Place, Chicago 38, Illinois.

NBS—National Bureau of Standards. Supt. of Documents, U. S. Gov't Printing Office, Washington 25, D. C.

NSA—Nuclear Science Abstracts, United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tenn.

PDA—Prevention Deterioration Abstracts, National Research Council, 2101 Constitution Ave., Washington 25, D. C.

RA—Refrigeration Abstracts, American Society of Refrigeration Engineers, 40 West 40th St., New York 18, N. Y.

RM—Revue de Metallurgie, Paris, France, 5 Cité Pigalle, Paris (9e), France.

RPI—Review of Current Literature Relating to the Paint, Colour, Varnish and Allied Industries, Research Association of British Paint, Colour & Varnish Manufacturers, London, Waldegrave Rd., Teddington, Middlesex.

TDD—Technical Data Digest, Air Material Command—Technical Service Section, Central Air Documents Office, Wright-Patterson Air Force Base, Dayton, Ohio.

TIME—Transactions of Institute of Marine Engineers, 85 The Minors, London EC 3, England.

UOP—Universal Oil Products, 310 South Michigan Ave., Chicago, Illinois.

ZDA—Zinc Development Association, Lincoln House, Turl Street, Oxford, England.



be formed in England, such as a section of NACE. However, geographical and other difficulties being considered, the Corrosion Group was thought best suited to Great Britain's needs.

One aim of the group is to make a career in the field of corrosion prevention more attractive to young scientists just starting their life's work, and to create opportunities for visits and exchange students and sponsoring of corrosion courses in universities.

## 1.2 Importance

### 1.2.2, 5.4.5, 5.9.3

**Economics of Industrial Maintenance Painting.** ROBERT R. PIERCE, Pennsylvania Salt Manufacturing Co., Philadelphia, Pa. Paper, Eighth Annual Conference, Natl. Assoc. Corrosion Engrs., Galveston, Texas, March 10-14, 1952. *Corrosion*, 8, No. 5, 178-182 (1952) May.

Data are based on results of a continuing research program, initiated by the Corrosion Engineering Products Department of the Pennsylvania Salt Manufacturing Company some eighteen months ago to determine factors and materials which promote maximum protective life in chem-industrial painting. This research includes 429 individual studies of the protective value of 62 maintenance painting compositions of 10 different basic types.

Determinations have all been conducted within the operating process areas of three major chemical manufacturing plants, widely divergent geographically, in fourteen different environments. Wire brushed, corroded and blasted surfaces have been examined. No artificial or simulated exposures were used in any phase of the program.

Where performance comparisons were required and in order to determine performance over surface irregularities normally encountered in maintenance painting, a new type test panel was used. The reliability of results from these panels was confirmed by 101 individual paint applications to actual operating plant equipment and structures. During the course of this program, actual and projected protective life expectancies of various painting systems and procedures were determined and converted to direct dollar cost and savings.

Some results from this program included in this article are:

1. Over 60 percent of the industrial maintenance paint compositions which by long experience have proved their adequacy in straight industrial use will fail rapidly when used for maintenance painting in a chem-industrial exposure.
2. Surface preparation by blasting will increase by two to four times the protective life of the maintenance paint system over wire brushed, corroded surfaces.
3. Maintenance paint system thicknesses below 5 mills are undependable in aggressive chem-industrial environments.
4. Regular schedules of paint inspection and repair can reduce cost of plant protection to the magnitude of 5 cents per square foot per year even in aggressive chem-industrial exposure.

## 1.3 Reviews

### 1.3.2.1.2

**Corrosion Tests and Service Performance.** F. L. LAQUE. INCO. Paper before

Am. Soc. Metals, 32nd Natl. Metal Congress and Exposition, Chicago, Oct. 23-27, 1950. Am. Soc. Metals, "Interpretation of Tests and Correlation with Service," Book, 1951, pp. 141-192.

Discussion of the types of corrosion tests and details of carrying them out. Laboratory tests, studies of the effects of corrosion products, salt spray, boiling nitric acid, plant, field and service corrosion tests, the API code for corrosion tests in petroleum refining processes, surface preparation, welding, stress, heating, cooling, and galvanic effects, measurements of corrosion damage, duration of tests and the combination of abrasion and corrosion are covered. Illustrations and 39 references are given.—INCO.

## 1.4 Bibliographies

### 1.4.2.3.6

**Bibliography of Electron Microscopy.** CLAIRE MARTON, SAMUEL SASS, MAX SWERDLOW, ALAN VAN BRONKHORST, AND HAROLD MERYMAN. National Bureau of Standards, Circular 502, Aug. 1, 1950, 87 pages.

Covers books and survey articles on instrumentation, electron optics, related instruments and applications prior to Jan. 1, 1950.—MR.

## 1.5 Directories of Materials

### 1.5.2.1.3

**Decontamination and Corrosion Resistance Properties of Selected Laboratory Surfaces.** C. D. WATSON, T. H. HANDLEY, AND G. A. WEST. U. S. Atomic Energy Commission. AECD-2996 (rev.), Aug. 29, 1950. 27 pp. (UF767 U3d.)

Fifty different materials were selected for testing and these were divided into 6 general groups as follows: Uncoated reference materials (for control purposes); baked interior panels; protective plastic coatings (air dried); laboratory bench-top materials; floor tiles; and disposable (strippable) plastic films.—BLR.

### 1.5.6.1, 6.2.1, 6.3.1

**Chemical Engineering Materials of Construction.** *Ind. and Eng. Chem.*, 41, No. 10 (1949).

**Aluminum Alloys.** E. D. VERINK, JR., AND R. H. BROWN. (*ibid.* 2095-2097).

A review of the applications and uses of aluminum alloys chiefly in industrial fermentation reactions, the manufacture of synthetic resins and fertilizer solutions and the storage and shipment of chemicals. 38 references.

**Wrought Copper and Copper-Base Alloys.** C. L. BULOW (*ibid.*, 2108-2114).—A summary of recent investigations on the properties, the fabrication and the corrosion-resistance of copper and its alloys. 117 references.

**Iron, Mild Steels, and Low-Alloy Steels.** C. P. LARRABEE AND S. C. SNYDER (*ibid.*, 2122-2124).—A summary of information published since 1948, with 27 references.

**Lead and Its Alloys.** G. O. HIERS (*ibid.*, 2124-2125).—A brief summary, with 25 references, of the uses of lead and its alloys in the chemical industries.

**Nickel and High-Nickel Alloys.** W. Z. FRIEND (*ibid.*, 2126-2132).—A review, dealing principally with nickel and alloys containing more than 40 percent

nickel or nickel + cobalt, in three sections: developments or improvements in alloys and studies of the physical properties of alloys; developments in the fabrication of nickel alloys including welding and forming; and developments in the application of the alloys including uses for high temp. and corrosion-resistance, particularly as applied to the chemical and process industries. 240 references.

**Stainless Steels and Other Ferrous Alloys.** M. H. BROWN AND W. B. DELONG (*ibid.*, 2139-2146).—A review covering published work which became available during the latter part of 1948 and early 1949. The paragraph headings are: passivity and surface films, corrosion, structure and mechanical properties, high-temp. behavior, welding, general, high-silicon irons, iron-nickel alloys, and austenitic manganese steels. 194 references.

**Tin and Its Alloys.** BRUCE W. GONSER (*ibid.*, 2147-2149).—A short review, similar to the preceding articles, with 44 references.

**Less-Common Metals.** H. H. UHLIG AND D. B. BROUGHTON (*ibid.*, 2153-2154).—A review of the literature over the past 10 years on the corrosion-resistance and other interesting properties of titanium, zirconium, tantalum, molybdenum, gold, platinum, and silver, with emphasis on recent trends and events. 67 references.

## 1.6 Books

### 1.6.2.3.9

**Bibliography of Electron Microscopy.** Book, edited by V. E. Cosslett. 8½ x 5½ in. Pp. 350. 1950. London: Edward Arnold and Co. (40s. net.)

Electron microscopy has become a well-organized craft which has been particularly fortunate in the publication of several valuable bibliographies of its literature.

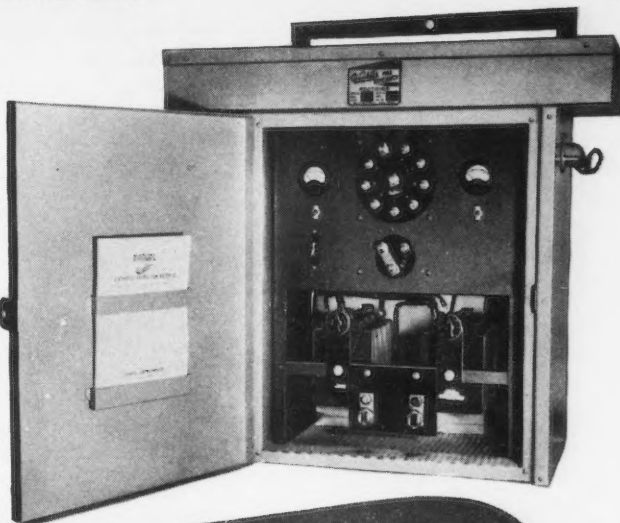
Much the most elaborate of these is now becoming available; this new bibliography covers the literature to the end of 1948, and, under the auspices of the Institute of Physics, quarterly supplements to it are being prepared and published in the "Journal of the Royal Microscopical Society."

The entries in the main bibliography and in each supplement are arranged in alphabetical order of the authors' names. Checking against previous bibliographies shows that approximately 80 percent of the papers then listed are included in the new effort. Just 40 percent of the 2200 references in the present volume carry abstracts, but no obvious system seems to have been used in choosing a particular type of paper for abstraction.

The bibliography is available printed on one side only of a series of sheets which may be cut up by each user and filed by any indexing system. The quarterly supplements are also to be printed on one side of the page. In addition, the main bibliography has been issued as a bound volume printed on both sides of the paper. In this volume also, the references are in alphabetical order of the authors' names and are not otherwise indexed. The exact purpose which this volume, which is the form submitted for review, is intended to serve is obscure, but to anyone who requires a complete and well-printed and bound collection of abstracts, here it is.—MA.

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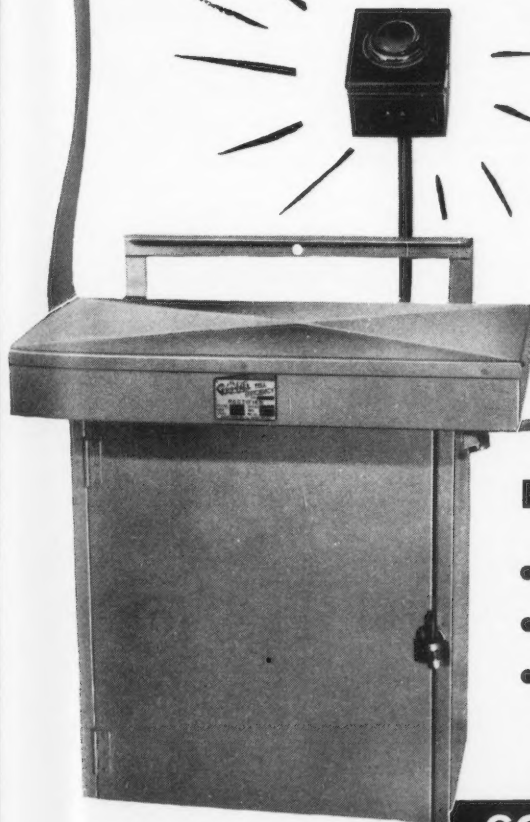
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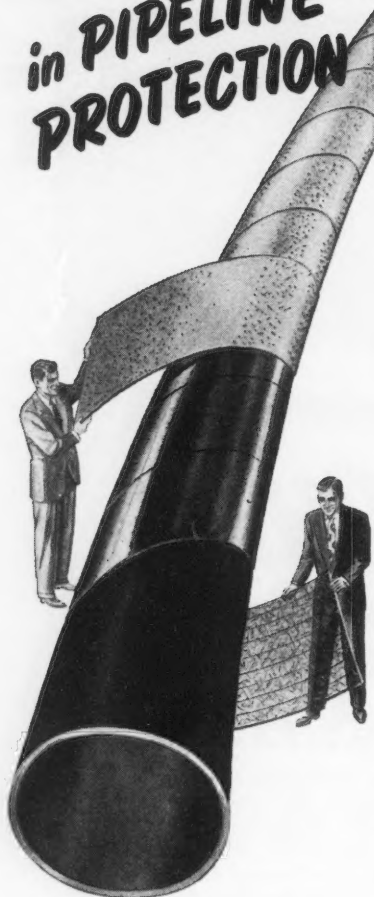
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## 2. TESTING

### 2.1 General

#### 2.1.2, 3.5.9

The Mechanism of Lubricating Oil Breakdown and Its Relation to Oil Corrosion. W. F. WEILAND. *Scientific Monthly*, 71, 121-122 (1950) Aug.

Four methods which have been used in the author's laboratory to correlate acid-vapor temperatures with incipient corrosion. (Acid-vapor temperatures are those at which volatile acids first form.)—MR.

### 2.2 On Location Tests

#### 2.2.3, 1.7.1, 2.1.2

Interpretation of Tests and Correlation With Service. Book, 1951, 198 pp. Am. Soc. Metals, Cleveland, Ohio.

A series of 4 educational lectures presented to members of the ASM during the 32nd National Metal Congress and Exposition, Chicago, Oct. 23-27, 1950. Refers exclusively to metals and alloys, their mechanical and corrosion testing, and service performance. Individual lectures are abstracted separately.—BLR.

#### 2.2.3, 1.7.1

Report of Subcommittee XV on Field Tests of Wire and Wire Products. (Report of ASTM Committee A-5 on Corrosion of Iron and Steel). ASTM Preprint No. 4, 1951, pp. 4-17.

Results of 14 year atmospheric corrosion tests on wires of bare steel; zinc, copper, and lead-coated steel; chromium- and chromium-nickel-steels.—BNF.

#### 2.2.4, 2.3.7, 7.1

Wear Tests and Service Performance. J. T. BURWELL, M.I.T. Paper before ASM, 32nd Natl. Metal Congress & Exposition, Chicago, Oct. 23-27, 1950. ASM Interpretation of Tests and Correlation with Service, 1951, 88-140.

Description of the principal types of wear indicating factors to be considered in evaluating service performance. Wear experiments are outlined and test and service experience in the wear of important types of machine elements given. Cutting, abrasive, galling, corrosive and minor types of wear, pitting fatigue, the correlation of wear measurements, the effect of hardness and wear test machines are discussed. Wear of a number of different steels, cast irons and alloys (Colmonoy, Nitralloy) are plotted against their hardness. They were run against the surface of case hardened molybdenum SAE steel. Service experience in the wear of journal bearings, cylinders, piston rings, gear teeth, lathe beds, dies, cutting tools, inspection fixtures and excavating equipment are included. Illustrations and 65 references are given.—INCO.

#### 2.2.6, 7.2

Corrosion of Metals Underground. *Gas Age*, 106, No. 4, 30-31 (1950) Aug. 17.

Outlines work of Denison and Romanoff of the National Bureau of Standards. Approximately 3000 specimens of representative pipe materials, exposed to soil arosion for periods up to 14 years, have been examined, and loss of weight and depth of pitting measured and inter-

preted. The specimens were buried at 15 test sites widely separated throughout the U. S.—MR.

### 2.3 Laboratory Methods

#### 2.3.1, 2.4.1, 1.6

Corrosion Testing Procedures. F. A. CHAMPION. 369 pages, 5½ x 8½ inches, cloth. 1952. John Wiley & Sons, Inc., 440 Fourth Ave., New York. \$6.25.

Author divides the subject into five parts: 1) Choice and preparation of metal and corrosive, 2) Exposure of the metal to environment in laboratory, field or service tests, 3) Cleaning of specimens preparatory to examination, 4) Examination of the specimens or the corrosive for the effects of corrosion, other measurements which indicate tendency to corrode and special tests, and 5) Expression and interpretation of results.

The introduction summarizes the general considerations of corrosion testing emphasizing the need for considering both the metal and the environment. In succeeding chapters by itemizing the factors entering into the tests and indicating to what extent variations in these factors may be expected to influence results, the author provides a lucid and easily read explanation of the procedures necessary.

Each chapter is followed by an extensive list of references, arranged chronologically. There is an alphabetical author index and a five page alphabetical subject index. Correlation of the data presented in the very large number of references alone is a substantial work.

#### 2.3.2, 6.5

Spray Testing With Natural and Synthetic Sea Water. Part I. Corrosion Characteristics in the Testing Metals. THOMAS P. MAY AND ALLEN L. ALEXANDER. *Proc. Am. Soc. Testing Materials*, 50, 1131-1143 (1950).

A formula is proposed for a solution which duplicates the inorganic components of natural ocean waters. Comparative tests were conducted in spray cabinets using 3 percent and 20 percent sodium chloride, natural ocean water, and the proposed solution. At the same time corrosion tests were conducted in natural environments. These tests reveal little similarity of performance between the natural environments and the spray cabinets using sodium chloride solution. The only exception exists in the deterioration of nickel-plated steel. In tests with ingot iron and plain steels, spray tests with natural and synthetic sea waters did not duplicate the types of corrosion usually observed in natural marine environments. Zinc oxide plated steel and solid zinc sheet corrode in a similar manner in synthetic and natural environments.—MR.

#### 2.3.2, 5.4.5

Present Position of Rapid Testing Techniques. G. ZEIDLER. *Farbe u. Lack*, 56, No. 11, 508 (1950).

A description of an accelerated breakdown test for paint films is given. The paint film, previously well dried at 40° C. is exposed to a high-power U.V. lamp for 1 hour, followed by immersion in water for 1 hour, then submitted to heat for 1 hour. This cycle is repeated three times a day; overnight, the film is stored at -5° C.—RPI.



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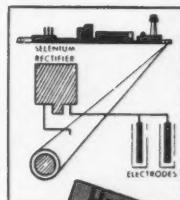
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## 2.3.4

**Symposium on Non-Destructive Methods of Testing.** Held at Constance, 8 Sept. 1950. *Metall.*, 4, No. 21/22, 469-470 (1950).

A short review of the contributions on: 1) radiographic, electrical-conductivity, inductive, and supersonic methods of crack detection; 2) the radiographic method of stress detection and 3) recent advances in the various types of induction instruments developed by Förster.—MA.

## 2.3.5

**Potentials of the Evolution of Hydrogen in Acid Solutions.** A. G. PECHERSKAYA AND V. V. STENDER. *J. Phys. Chem. (USSR)* (*Zhurnal Fizicheskoi Khimii*), 24, 856-859 (1950).

The potentials of the evolution of hydrogen were determined from a 2 normal

solution of sulfuric acid at 25° C using current densities of 10-2000 amp./sq. m. for zinc, cadmium and other metals.—ZDA.

## 2.3.5, 6.3.1, 4.3.2

**Irreversible Electrode Potentials of Metals in Solutions of Hydrofluoric Acid.** (In Russian). M. M. KURTEPOV AND A. S. FEDOSEEVA. *Doklady Akad. Nauk S.S.S.R.* (Reports of the Academy of Sciences of the USSR), 75, No. 4, 563-564 (1950) Dec. 1.

Gives chart showing electrode potentials of 25 metals after 1 minute and 24 hours in 1.0 normal, 0.1 normal, and 0.01 normal hydrofluoric acid.—BNF.

## 2.3.5, 5.2.3, 4.5.2

**Potential Measurements for Determining Cathodic Protection Requirements.**

SCOTT P. EWING. Paper, Seventh Ann. Conf., Natl. Assoc. Corrosion Engrs., New York, N. Y., March 13-16, 1951. *Corrosion*, 7, No. 12, 410-418 (1951) Dec.

An investigation has been made for the purpose of determining if buried or submerged steel pipe can be supplied adequate cathodic protection against corrosion by the maintenance of some constant open circuit potential. The experimental work consisted in establishing the open circuit potentials required to prevent corrosion of test pipe specimens exposed under a variety of conditions. Identical groups of six specimens each were exposed in four different environments. Test locations were selected to include as wide a variation in soil conditions as possible and one group of specimens was exposed in water.

Current was applied to five of the six specimens in each test group with the object of maintaining the open circuit potential of each specimen at a designated value. Measurements were made throughout the test of the open circuit potential and the amount of current supplied to each respective specimen. At the conclusion of the four tests, which were run for some 70-80 days, actual corrosion weight losses for all test specimens were determined and then correlated with the current and potential measurements. The results are presented and the data are explained and illustrated by accompanying graphs and tables.

These data indicate that in each test environment there was a definite potential at which corrosion was stopped. This potential was found to vary with the environment, and in all cases, was less than -0.85 volt with reference to the copper-copper sulfate electrode. The test results also indicate that films, or coatings, formed by the applied current are of considerable importance in some soils in reducing current requirements, and that there is a definite advantage in applying sufficient current to form the coating as soon as possible after the cathodic protection installation is completed.

In appraising the test method used in this investigation the conclusion is reached that the method is apparently sound and that it can be used to determine what must be done to the pipe potential in order to prevent corrosion in any environment. The method appears to be capable of more precise results and recognition is made of the fact that further development and refinements are desirable. In this respect certain improvements are suggested for consideration.

## 2.3.6

**Periodic Phenomena in the Corrosion of Metals By Vapors.** (In French.) R. DUBRISAY. *Bull. societe chimique France*, 1058-1060, Nov.-Dec. (1950).

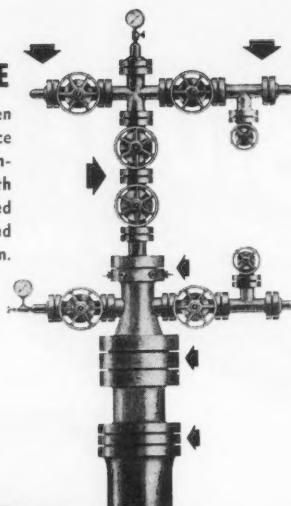
When a thin sheet of silver or copper with a small hole in it is placed over a test tube containing a fragment of iodine, concentric colored rings form around the hole, due to decreasing thickness of iodine formed at increasing distances from the hole. A quantitative study was made of this phenomenon for various periods of time and temperature up to 70° C. Effects of various factors are thoroughly analyzed. Data are tabulated and charted.—BLR.

## 2.3.6, 3.2.2

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Corrosion in the ring is certain to result when dissimilar metals are flanged up in the presence of a low resistance liquid or electrolyte. Machining of cloth-reinforced plastics exposes the cloth to liquids which hasten deterioration. Forged steel ring gaskets cannot stop this corrosion and alloys frequently increase the rate of corrosion. But it can be eliminated!



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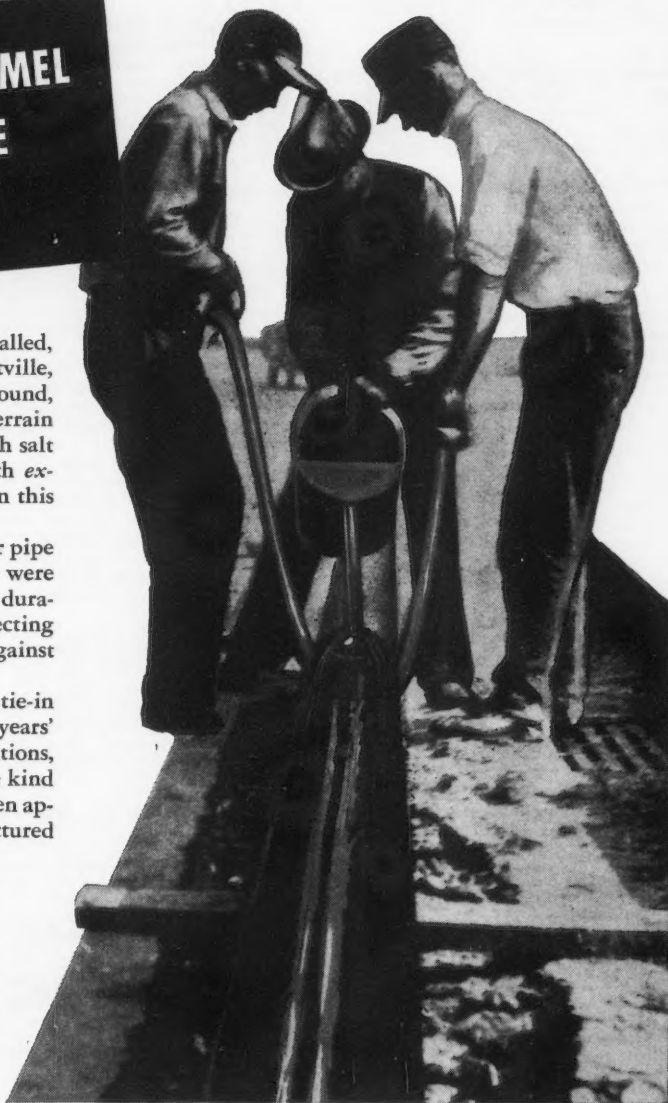
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IN 1926, a 6-mile, 8-inch gas pipe line was installed, extending from Atlantic City to Pleasantville, N. J. This line was laid about two feet underground, in land known locally as "the meadows." The terrain is tidal marsh and high tides flood the land with salt water. No steel pipe line, unless provided with exceptional protection, could possibly last long in this corrosive soil.

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In 1951, a pipe section was removed so that a tie-in could be made with another pipe line. After 25 years' exposure to tidal marsh and sea-water conditions, there wasn't a sign of corrosion. This shows the kind of protection Bitumastic Enamel gives, even when applied by the old-fashioned manual method pictured here.

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Photograph made in 1926 shows gas pipe line (Atlantic City—Pleasantville) being coated with Bitumastic Enamel. A single coat of enamel was applied by the "Sling Method" to a minimum 1/16" thickness. In 1951, a pipe section was removed so that a tie-in with another line could be made. An examination revealed no sign of corrosion.

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### Cracks in Metals, *Materials Methods*, 31, No. 4, 4, (1950) Apr.

Surface flaws and cracks in metals are rapidly detected by an "oil-powder" at the Naval Engineering Expt. Station, Annapolis. A film of penetrating oil is first applied to the surface being inspected, then removed, and the area dusted with nonabrasive detecting powder. The defects show up in red against a white background.

### 2.3.6, 2.6

**Replica Techniques in Electron Microscopy.** A. W. AGAR AND R. S. M. REVELL. *Brit. J. Applied Physics*, 2, No. 1, 8-11 (1951) Jan.

How to obtain clean and reliable replicas for the study of metal surfaces under comparatively low magnification (X 2,000). Washing; mounting; removing particles by successive film stripping. Modified replica processes; shadow-casting and preshadowing.—BNF.

### 2.3.7, 3.5.9, 6.2.2

**Corrosion—A New Mechanism for Measuring the High Temperature Oxidation of Iron has been Proposed as a Result of a Metallographic Investigation.** M. G. FONTANA. *Ind. & Eng. Chem.*, 43, No. 4, 71A-72A+ (1951) April.

New mechanism for measuring the high temperature oxidation of iron has been proposed as a result of a metallographic investigation. A chromium oxide distemper was applied by dipping iron specimens in a slurry of chromic oxide. The coating was allowed to dry and then the specimens were oxidized. In these tests the chromic oxide remained in contact with the iron surface, indicating practically no diffusion of oxygen into the iron. The distemper coating also permitted observation of blisters which form in the oxide. On oxidation of small specimens it is to be expected that the resultant structure would be asymmetrical and contain voids of considerable size. A large specimen oxidized for a sufficient time will exhibit no adherent or continuous oxide in contact with Photomicrographs.—INCO.

### 2.3.7, 2.4.3, 5.4.5

**Magnet Thickness Gauge.** *Product Finishing* (London), 3, No. 12, 61 (1950).

A small non-electric magnetic thickness gauge for measuring the thickness of non-magnetic materials on steel surfaces has a range of 0.0002-0.010 inches and an accuracy of  $\pm 15$  percent.—RPI.

### 2.3.7, 7.8

**Corrosion of Thin Wires. I.—Corrosion of German Silver, Eureka, Copper, and (Armco) Iron in Sea-Water.** (In English.) HIROSHI YOSHISAKI. *Science Reports of Research Inst., Tohoku Univ.* (A), 2, No. 1, 96-101 (1950).

The corrosion of nickel silver, Eureka, copper, and Armco iron in sea water at  $25 \pm 1^\circ \text{C}$  was studied by measuring the elongating of the wires under load after various periods of immersion in sea-water. The results do not conform with anticipations based on the assumption of homogeneity of internal structure of the wires, and are attributed to inhomogeneity of distribution of chemical singularities in the wires.—MA.

### 2.3.9, 2.4.3

**Introduction to Symposium on Ultrasonic Testing.** H. G. AMTSBERG. A.S.T.M. Symposium on Ultrasonic Testing, 1951, pp. 1-2.

The author states that: 1) ultrasonic testing is used extensively; 2) it is practical, and is applied intelligently in many industries; 3) it is not just another device for rejecting materials, but aids manufacturing; and 4) standard equipment that can be used by non-technical personnel is available.—MA.

### 2.3.9, 2.3.7, 5.4.5

**New Technique for Measuring Paint.** *Science News Letter*, 58, No. 21, 335 (1950).

X-ray techniques for the study "inter alia" of paint film thickness and corrosion were described at a New York Academy of Sciences symposium.—RPI.

### 2.4.3, 1.2.2

**Symposium on the Role of Non-**

**Destructive Testing in the Economics of Production.** Presented at the Fifty-Third Annual Meeting, American Society for Testing Materials, Atlantic City, N. J., 27 June 1950. (Special Technical Publication No. 112.) Med. 8vo. Pp. (v)+157, illustrated. 1951. Philadelphia 3, Pa.: Obtainable from The Society, 1916 Race Street, Philadelphia 3, Pa.

Contents: 1) Robert C. McMaster and Samuel A. Wenk, "A Basic Guide for Management's Choice of Non-Destructive Tests"; 2) Kent R. Van Horn, "Discontinuities in Cast and Wrought Products That Can Be Revealed by Non-Destructive Tests"; 3) John W. Juppenlatz, "The Role of Non-Destructive Testing in the Economics of Castings"; 4) W. L. Schwinn, "The Economics and Practical Application of Cobalt 60 in the Radiographic Inspection of Steel Weldments"; 5) C. D. Moriarty, "The Economics of Wrought Steel Inspection"; 6) L. W. Ball, "Management's Responsibility for Insistence on Non-Destructive Testing in the Development of New Engineering Products and Processes."—MA.

## 2.4 Instrumentation

### 2.4.3, 2.4.2

**New Instruments for Internal Inspection of Tubing.** *Eng. and Boiler House Rev.*, 66, 59-60 (1951) Feb.

Two instruments, which have recently become available in the United States, the "Penetron" and "Probolog", are specifically designed to assist in the inspection of pipelines and tubes 'in situ' and by nondestructive methods. The "Penetron", utilizing gamma radiation, can be used to check the thickness of tube or pipe walls and thus detect any tendency to thinning during service; it does not, however, detect the presence of scale or pits and holes in the tube metal. The "Probolog", operating on electron-magnetic principles, is capable of detecting holes, pits or other forms of wastage in tube walls and was specially developed for checking the tubes of heat exchangers.—TIME.

### 2.4.3, 5.4.5

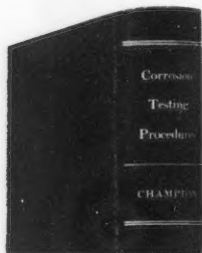
**Paint Testing Instruments.** L. A. COOPER. Sawell Publications Ltd., London, 1950, 72 pp. Price 7/6.

A collected reprint of a series of articles published in *Product Finishing* (cf. *Review*, 1950, 295, 505) dealing with the principles of measurement of consistency, gloss, reflectance, hiding power, drying time, film thickness, abrasion and weathering resistance in terms of apparatus commercially available. The discussion is in general sound, but it is thought that the main value of the book will be in acquainting prospective purchasers about equipment available rather than in assisting in choice between different instruments.—RPI.

### 2.4.3, 2.3.9

**Measurement of Surface Smoothness.** HENRY L. KELLNER. *Proc. Am. Electroplaters' Soc.*, 37, 105-124 (1950).

A review of methods available. These include microscopic methods, the use of the micrometer, the use of electro-mechanical instruments such as the Profilometer and Brush Surface Analyzer, acoustical methods, replica techniques, the use of the electron microscope, and methods involving reflectance. 23 references.—MA.



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TYGON sheeting is made in thicknesses ranging from 1/64" to 1/2" and in continuous lengths or sheet form depending upon thickness. The main use for TYGON sheeting is in the lining and covering of such equipment as tanks, tank covers, diffusional towers, vats, bins, hoppers, troughs, blowers, impellers, fume hoods, and fume ducts. It is also die-cut into gaskets for positive seals in all types of processing equipment.

As sheeting, TYGON resists chromic acid in concentrations up to 30% and at temperatures up to 130°F. At room temperature, the concentration limit may be raised to 50%. If a mixture of chromic and sulfuric acids is used, the maximum concentration should be 15-20% at 130°F. Against a hard chrome plating solution, TYGON should be used with a protective sheathing of acid resistant brick and cement. Such a sheathing also permits the use of TYGON against other solutions of chromic acid at service limits considerably above those for an unprotected lining. Higher service limits are also possible when TYGON is used as gasketing, since only a small portion of the gasket is usually exposed.

In contact with chromic acid, TYGON undergoes some physical changes. Prolonged exposure causes surface hardening and a noticeable color change. However, these changes do not affect TYGON's protectability.

As molded goods, TYGON has many uses—uses limited only by the size and shape that

can be imparted to a thermoplastic material by mold and press. When molded, TYGON exhibits the same resistance to chromic acid as it does in sheet form. In some cases, higher service limits are possible depending upon the size, design, and use of the molded item.

A major use of TYGON is as extruded tubing and piping. The full flexibility, glass-like clarity, lightweight, smooth surface, and high strength of TYGON tubing has done much to simplify many piping jobs in the laboratory and the plant. Extruded solid cord and channel also find use as gasketing, expansion jointing and packing.

In extruded form, TYGON also resists chromic acid in concentrations up to 30% and at temperatures up to 130°F. At room temperature, the concentration limit may be raised to 50%. Against mixtures of acids, service limits should be lowered to a maximum of 20% at 130°F. On prolonged exposure to chromic acid the tubing tends to stiffen, harden and discolor. However, the resistance and functioning of the tubing is not affected. Because of the staining that results, clear TYGON tubing is not recommended for use with chromic acid.

TYGON, as a paint, protects all types of equipment, structural steel, walls, and ceilings against the fumes and spillage of chromic acid. Special consideration should be given to the physical limits of a thin film. TYGON paint should not be used as a lining material. In all exposures to chromic acid, a minimum system of a primer plus five top coats of TYGON paint is recommended.

Properly applied, TYGON paint resists the fumes and spillage of chromic acid in any concentration and up to 200°F. Spillage should be washed away as soon as possible. Some staining from spill is to be expected.

As a plastisol (TYGOFLEX), TYGON finds use as a heavier duty coating and in the casting or "slush" molding of flexible parts and fittings. In this form, the resistance to chromic acid depends somewhat upon the thickness of the coating deposited, but approximates the resistance of TYGON sheeting.

Although its use against chromic acid is more limited than it is against other chemicals, properly applied TYGON provides low cost protection and a high degree of safety in handling this acid and related chemicals.

*In addition to TYGON in its various forms, we also manufacture a number of other materials capable of handling chromic acid in any concentration and under all types of operating conditions. These products include chemical stoneware and porcelain, acid proof brick and cements, homogenous lead linings, and other organic linings and coatings.*

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5-D

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ENGINEERS, MANUFACTURERS, ERECTORS OF CORROSION-RESISTANT EQUIPMENT SINCE 1865

## 2.4.3

**The Development of a Flaw Detector for Tubes.** R. D. KODIS. *Proc. Am. Soc. Testing Materials*, 50, 1196-1206 (1950).

The a.c. bridge method of magnetic flaw detection was evaluated by application to smooth-bore tubes. Practical difficulties with the method led to investigation of the induction method. By proper combination of detector-head design and a recorder capable of displaying three variables, cracks are shown in picture form for easy evaluation. Use of the induction method for inspecting gun tubes, and for production inspection. It is demonstrated that the induction method has greater speed, reliability, definition and sensitivity than present methods for inspecting gun tubes and can be engineered into a production inspection tool.

## 2.4.2, 8.9.3

**Mobile Radio Units Help Pipe Line Corrosion Survey Work.** *Pipe Line News*, 22, No. 9, 26 (1950) Sept.

Experience of Standard Oil Co. (Ind.) pipe line corrosion survey parties in using two portable transmitters and receivers in their survey work is described.

## 2.4.3, 2.3.6, 7.1

**Red Means Danger.** REBECCA H. SMITH. *Western Machinery and Steel World*, 41, 70-72 (1950) Feb.

"DPM" (dye penetrant method) for surface-defect detection, developed and used on gas-turbine parts.—MR.

## 2.4.3, 2.3.6

**Low-Cost Inspection Method Finds Metal Surface Flaws.** *Steel*, 127, 68, 70 (1950) July 24.

Dye-penetrant technique which em-

ployes inexpensive, portable equipment to detect minute surface openings in ferrous and nonferrous metals.—MR.

## 2.4.3

**Ultrasonics and Their Use for Non-Destructive Testing of Materials.** (Concluded.) N. G. NEUWEILER. *Microtechnic*, 4, 60-66 (1950) Mar.-Apr.

Basic operating principles of the Hughes ultrasonic flaw detector, as well as the actual equipment, including the filter system. Typical results on steel forgings.—MR.

### 3. CHARACTERISTIC CORROSION PHENOMENA

#### 3.2 Forms

## 3.2.2

**Some Factors Affecting the Rate of Diffusion of Hydrogen Through Steel During Electrolytic Pickling.** W. A. BELL, G. J. METCALFE AND A. H. SULLY. Paper before Electrodepositors' Tech. Soc., Torquay, England, April 12, 1951. *J. Electrodepositors' Tech. Soc.*, 27, 1951, Adv. Copy No. 2, 18 pp.

Description of the measurements which were made of the diffusion of hydrogen through mild steel and through steel to composition DTD4A during cathodic treatment in various  $H_2SO_4$  and  $NaOH$  electrolytes. The apparent hydrogen diffusion varies over wide limits between various electrolytes. For mild steel, the effect of cold work on diffusion rate differs between acid and alkaline electrolytes. The variations in diffusion rate are ascribed to the for-

mation of cathodic films which affect the rate of transfer of hydrogen atoms into the metal lattice. The quantity of hydrogen required to cause embrittlement of steel is very much less than that required to saturate the steel, 16 references.—INCO.

## 3.2.2

**Influence of Amplitude and Rate of Plastic Deformation on Diffusion of Hydrogen in Iron and Steel.** (In French.) PAUL BASTIEN AND PIERRE AZOU. *Compt. Rend.*, 232, 69-71 (1951) Jan.

The above was investigated using specimens saturated with hydrogen by 48 hours exposure to 10 percent hydrochloric acid + 0.01 percent sodium sulfide in water. A tensile test was applied for periods varying from 0.07 seconds to 2 hours, 20 minutes. In the absence of preliminary plastic deformation, hydrogen had little effect.—BLR.

## 3.2.2, 6.2.5, 6.3.5, 6.3.13

**Interchangeability of Cb and Ta in Type 347 Stainless.** W. O. BINDER. *Metal Progress*, 59, 219-227 (1951) Feb.

Studies were made on 18-8 steels containing 0.035-0.07 percent carbon using tantalum metal, the ferrocolumbium alloy containing approximately 55 percent columbium and 5-7 percent tantalum, and a new ferroalloy containing about 40 percent columbium and 20 percent tantalum. Object was to determine the equivalency of tantalum with respect to columbium in preventing intergranular attack, the probable ratio of columbium plus tantalum to carbon required for practical immunity to intergranular attack, and the effect of a partial substi-

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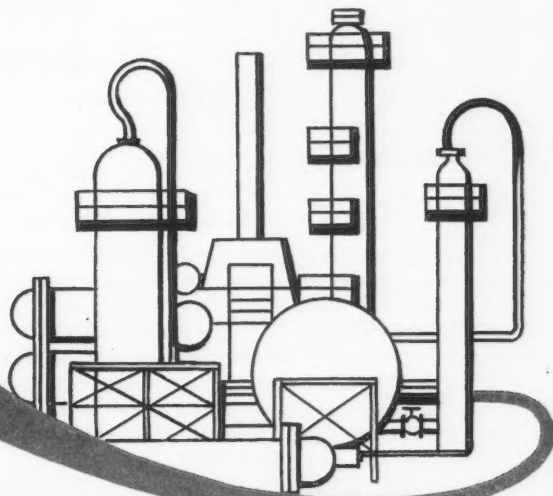
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tution of tantalum for columbium on the strength and ductility of normal columbium-bearing 18-8 steel at room and elevated temperatures. Test media were boiling 65 percent nitric acid, 10 percent nitric acid + 3 per cent hydrofluoric acid at 70° C., and boiling copper sulfate-sulfuric acid solution. Results indicate that twice the weight of tantalum will give protection against intergranular attack approximately equal to that of columbium. On an equal-weight basis, tantalum is as effective as columbium for promoting high-temperature strength.—BLR.

### 3.2.2, 6.3.7

Some Mechanical Properties of Graphite at Elevated Temperatures. C.

MALSTROM, R. KEEN, AND L. GREEN, JR. *J. of Applied Phys.*, 22, 593-600 (1951) May.

Short-time tensile breaking strength of various grades of graphite was measured as a function of temperature from room temperature to the sublimation point. It was found that strength approximately doubles in going from room temperature to about 2500° C and then decreases rapidly to zero near the sublimation point. Graphite can be used as a structural material at temperatures much higher than those permissible with other materials. Creep characteristics of grade ECA graphite under tensile stress at elevated temperatures were investigated. Low-frequency dynamic measurements of Young's modulus of this graphite at 1000-2000°C showed an in-

crease of the modulus with temperature. 15 references.—BLR.

### 3.2.2, 5.3.4, 6.3.10, 6.3.14, 6.3.8

Hydrogen Embrittlement in Nickel, Tin and Lead Electroplating. C. A. ZAPPE AND M. E. HASLEM. *Plating*, 37, 610-613 (1950) June.

Quantitative measurements are reported for brittleness of mild-steel and low-carbon stainless-steel wires electroplated with nickel, tin and lead. Typical baths provided data for an evaluation of the scope of common hydrogen problems, such as brittleness and blistering, likely to be encountered. The standardized procedure used permits the comparison of a number of chromium, cadmium, zinc, copper, nickel, tin and lead plating processes.—MR.

### 3.2.2, 5.3.4, 6.3.2, 6.3.19

Hydrogen Embrittlement in Cadmium and Zinc Electroplating. C. A. ZAPPE AND M. E. HASLEM. *Plating*, 37, 366-371 (1950) Apr.

Embriement through hydrogen absorption by the cathode is measured in terms of bend angle sustained by plated wires before fracturing. Both mild steel and stainless steel were studied. Results provide a basis for predicting the scope of the hydrogen problem that might be encountered on changing from one plating operation to another, as well as a quantitative measure of hydrogen injury caused by each of the baths studied.—MR.

### 3.2.2, 6.2.5, 7.4.2

A Case History Involving Intergranular Failure of Stainless Steel Heater Tubes. G. A. WORKS. *Oilum Refinery*, Union Oil Company of California, Redwood, California. Paper; San Francisco Bay Area Section, Natl. Assoc. Corrosion Engrs., September 12, 1951. *Corrosion*, 6, No. 6, 217-221 (1952) June.

Failure of a number of Type 316 stainless steel tubes in a fired heater at a phenol solvent treating plant after ten years of service is outlined. Metallurgical investigations showed that carbide precipitation at the grain boundaries had occurred after this length of service at a temperature of about 750 degrees F thus sensitizing the tubes to intergranular corrosion. Inspection of the remaining tubes in the heater by a Probolog survey and by fluorescent dye penetrant showed that approximately two-thirds of the tubes while in a susceptible state had not yet progressed to the point of developing cracks.

The possibility of salvaging these uncracked tubes for further use was investigated. Samples were heat treated at 1600 degrees F for 24 hours. Microscopic examination of polished and etched specimens showed a definite agglomeration of the carbides at the grain boundaries tending to break up the network of precipitates. Tensile and bend tests on heat treated and welded specimens showed good results.

This heat treatment was carried out on the uncracked tubes and they were re-installed in the heater. No further difficulty has been encountered after 24 months of service.

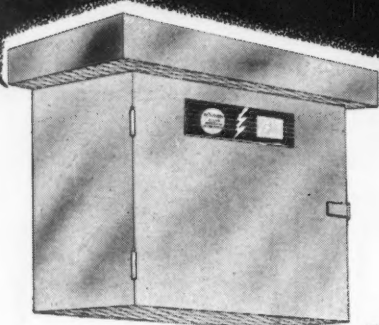
### 3.2.2, 6.3.19

The Pitting of Zinc By Distilled Water and Dilute Solutions. U. R. EVANS AND D. E. DAVIES. Department of Metallurgy, University of Cambridge, Cambridge, England. Reprinted from the *Journal of the Chemical Society (U.K.)*

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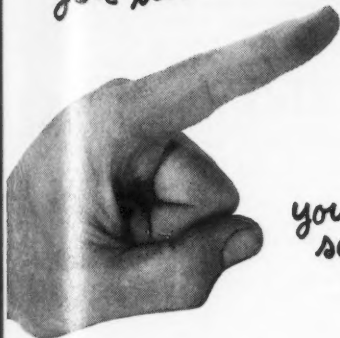
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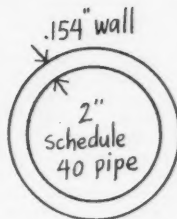
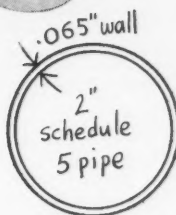
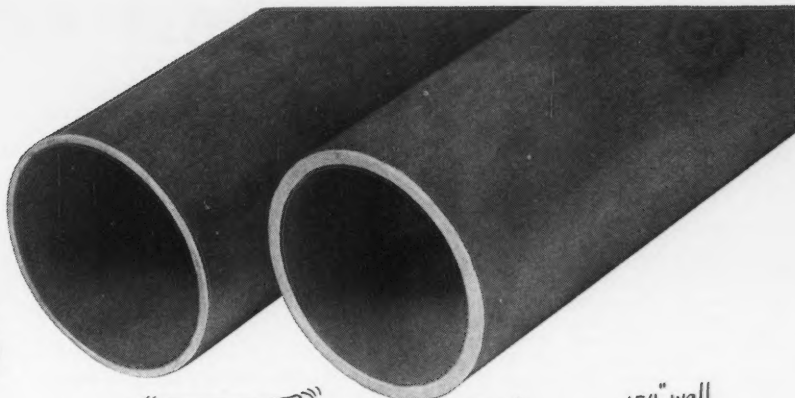
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## STAINLESS TUBING & PIPE



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1951. EDITOR'S NOTE: This paper was reprinted in Corrosion because it shows how valuable information can be obtained by simple experimental techniques. *Corrosion*, 8, No. 5, 165-170 (1952) May.

The object of the research was to explain the vertical arrangement of pits produced on zinc by distilled water, to study the corrosion products and to explain their formation. The vertical alignment is due to corrosion product from an upper pit lodging at points below and screening them from oxygen; lines of pits can also be set up by the screening action of a polythene thread. A zinc disc whirled in distilled water containing oxygen suffers no pitting. In stagnant water containing oxygen under high pressure, zinc remains unchanged; at ordinary pressure, it becomes pitted. The pits contain white matter and are surrounded by white rings (oriented zinc oxide and some  $\beta$ -hydroxide); further from the pits are films of interference-color thickness (also orientated zinc oxide), whilst negatively charged colloid particles appear in the liquid. Salt solutions produce a distribution of attack different from that given by distilled water. The results are explained on the theory of oxidation and corrosion advanced by Hoar and Evans.

### 3.2.2, 6.2.3

**Hydrogen Embrittlement of SAE 1020 Steel.** J. B. SEABROOK, N. J. GRANT AND DENNIS CARNEY. *J. Metals*, 188 (1950), Nov.; *Trans. Am. Inst. Mining Met. Engrs.*, 188, 1317-1321 (1950).

Hot-rolled SAE 1020 machined into tensile-test bars was cathodically charged

with hydrogen. Vacuum tin-fusion analysis provided accurate values of hydrogen. True stress-true strain tensile tests with and without aging of the test bars and at various strain rates indicate the role of hydrogen as an embrittling agent. 13 references.—MR.

### 3.2.2

**Action of Corrosion and Stress on 13% Cr Stainless Steel.** H. H. UHLIG. *Metal Progress*, 57, 486-487 (1950) Apr.

Experiments which suggest that failure by cracking of stressed martensitic stainless steels exposed to a corrosive environment under stress may be best described as "hydrogen embrittlement." —MR.

### 3.2.2

**Why Machine Parts Fail. III.—Modes of Failure. IV.—Fatigue Fractures.**

CHARLES LIPSON. *Machine Design*, 22, No. 7, 141-145; No. 8, 157-160 (1950);—*Cf. ibid.*, Nos. 5 & 6, 95-100, 111-116 (1950).

III.—Fracture may show the characteristics of ductile or shear type of failure or may be of the more normal brittle type. Lipson discusses the types of failure to be expected under certain service conditions and lists the factors affecting the mode of failure. These factors, discussed in detail, include manner of loading, the type of stress, speed of loading, stress direction, stress distribution, temp. conditions, the period of load application, the type of material, and surface treatment.

IV.—Lipson stresses that the interpretation of fractures may frequently be very difficult, as the appearance of each can be influenced by many variables. Certain basic types of fatigue fracture

are common, these being illustrated and discussed with details given of their mode of formation and propagation. All fatigue fractures occur without any perceptible ductility and always show the distinct zones of fatigue and instantaneous fracture. Stop marks are usually found which indicate the rate of progress of the fatigue crack.—MA.

### 3.2.2

**Corrosion Cracking.** U. R. EVANS. Institution of Metallurgists. "The Fracture of Metals," pp. 68-100. Book, 1950, 138 pp.

Chemical, electrochemical, crystallographic and stress corrosion. Theoretical picture with some practical examples. Suggests methods of prevention. Data for aluminum, copper, nickel and their alloys and for carbon, alloy and stainless steels, includes micrographs. 47 references.—MR.

### 3.2.3, 3.4.6, 2.3.4

**Simple Test for Detecting the Porosity of Films.** H. FREYTAG. *Chem.-Zt.*, 74, No. 37, 551 (1950); *Bull. Inst. Paper Chem.*, 21, No. 3, 185 (1950).

Hydrogen peroxide (35-40 percent) is poured into a small beaker with a wide opening; the beaker is closed with a clean cork stopper with a central boring. A strip of the film to be tested is placed over the opening and a lead sulfide reagent paper of the correct size on top of it; both are pressed down with a clean glass plate. After some time, according to the thickness and porosity of the foil, the evaporating hydrogen peroxide will cause a brightening of the dark brown color of the reagent paper. The degree of brightening under identical conditions of temperature, diameter of cork boring,

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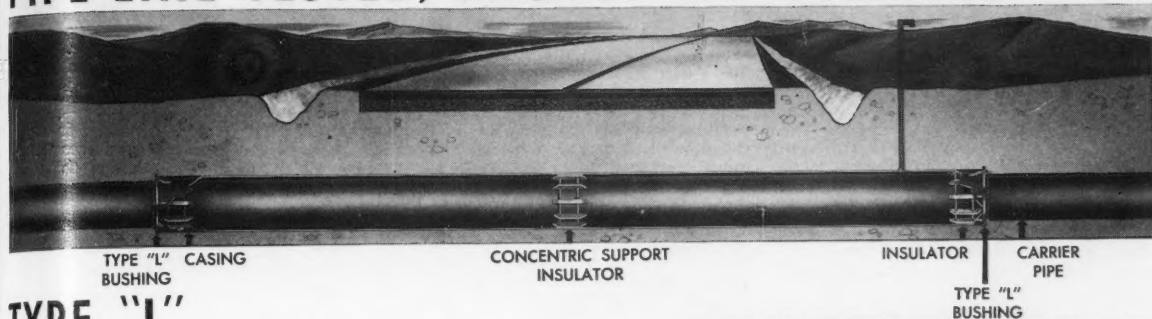
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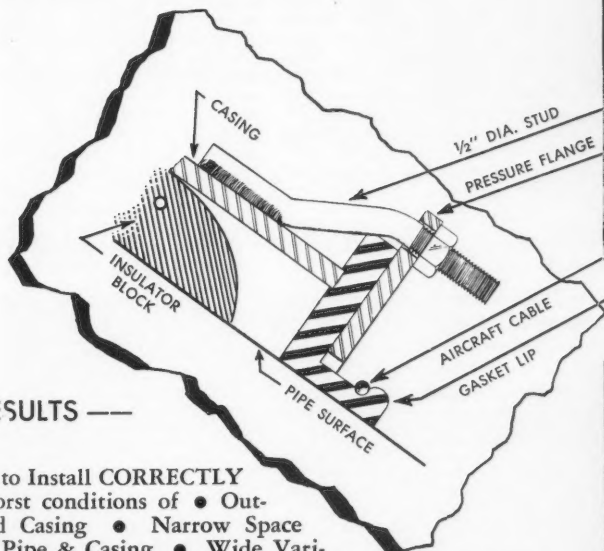
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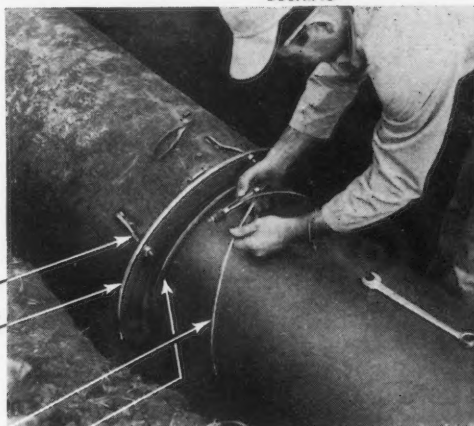
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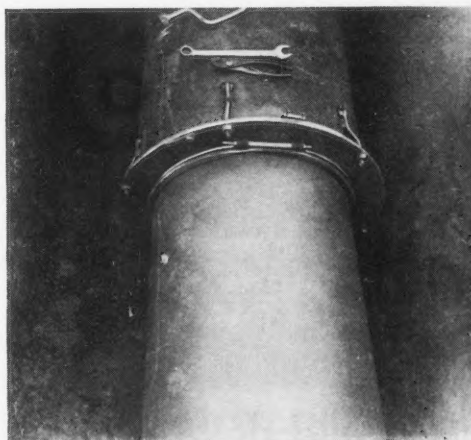


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  - BUTTED AGAINST CASING as pressure flange is tightened on 1/2" studs welded to casing.
  - TIGHT TO THE PIPE as aircraft cable (4,600# tensile) is tightened to gasket lip. Union is rubber covered to INSULATE galvanized cable assembly from bare casing structure.
3. Always Insulated from the pipe line when a Concentric-Support Insulator is installed just inside casing after the "drag section" is in place.



Assembling cable with rubber-covered union for clamping gasket lip to pipe.



THE COMPLETED INSTALLATION  
Note that galvanized cable and union are insulated from pressure flange and from pipe.

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TULSA 9, OKLAHOMA

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and reaction period permits the evaluation and comparison of the porosity of different foils. In a modification of this method recommended only for very thin and porous films, the surface of a filter paper is charged with hydrogen peroxide vapor; the foil is placed upon this side, followed by the reagent paper, and the three layers are pressed between two clean glass plates. The results are comparable only when the pressure is identical in all experiments.—RPI.

### 3.3 Biological Effects

#### 3.3.4, 7.2

**Aerobic Microbiological Corrosion of Water Pipes.** Parts I and II. ERIK OLSEN (De Danske Sukkerfabrikker's Centrallaboratorium, Copenhagen), AND WACŁAW SZYBALSKI (Wyeth Inc., Philadelphia). *Acta Chemica Scandinavica*, 3, 1094-1116 (1949); *Corrosion*, 6, No. 12, 405-414 (1950) Dec.

Tubercles in water pipes contain, in addition to large quantities of amorphous ferric hydroxides, iron bacteria

surrounded by sheaths. Although these bacteria are not the direct cause of corrosion, they oxidize ferrous ion to ferric ion, and thereby gain the energy necessary for the assimilation of carbon dioxide. Their sheaths assist in the formation of a membrane that is relatively impervious to oxygen, and, in the process, decrease the quantity of oxygen in the tubercle vicinity, thus establishing a micro-electrochemical cell. With increasing membrane thickness, the inside of the tubercle becomes more anaerobic, the difference in potential between the iron surface underneath and outside the tubercle increases, and corrosion accelerates. Once the differential cell has formed, corrosion (pitting) proceeds practically independently of the metabolic activity of the bacteria.

The primary formation of microgalvanic cells by iron bacteria and the mechanical reinforcement of the tubercles by the bacterial sheath was proved in experiments using differential aeration cells; these cells were obtained by employing germ-filtered water with and without inoculation with iron bacteria. The average increase of corrosion caused by these bacteria was about 10 percent.

Inhibition of further corrosion was attempted by changing the equilibrium of the water in such a way that a thick layer of chalky rust formed on the tubercle, thereby decreasing the current density on the anodic areas. Chalky rust layers can be formed by passing the water through freshly filled magnofilters.

Addition of chlorine to the water does not kill bacteria, but may suppress the formation of new bacterial colonies.

### Positions Wanted

**Graduate Metallurgical Engineer** desires position as corrosion engineer. Nine year's experience in automotive and pipeline field. Write CORROSION, Box 52-9.

**Corrosion Engineer**, 8 years' pipe line experience desires sales position as representative for manufacturer of pipe line or cathodic protection equipment. Write CORROSION, Box 52-11.

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## PAINT CHEMIST OR PAINT TESTER

Degree in chemical engineering or chemistry preferred, but not essential with compensating experience in paint laboratory on panel testing, formulation service testing, and/or application problems. Write giving qualifications and experience to:

EMPLOYEE RELATIONS DEPARTMENT  
PHILLIPS PETROLEUM COMPANY  
BARTLESVILLE, OKLAHOMA

### 3.4 Chemical Effects

#### 3.4.3, 5.2.1

**The Corrosion Products of Zinc Anodes Used Underground.** E. A. ANDERSON. *Corrosion*, 6, 129-131 (1950) Apr.

The presence of sulfates, such as gypsum, in the soil around zinc anodes used in the cathodic protection of pipelines has been observed to largely prevent the loss of current flow normally found in ordinary soils. Examination of the corrosion products on two anodes used in gypsum-bearing soil indicated the mechanism by which sulfates affect film resistance.

#### 3.4.6

**Metallic Corrosion and Absorption.** K. WICKERT AND J. KROLL. *Werkstoffe u. Korrosion*, 1, 105 (1950) Mar.

Statement required by the Act of August 24, 1912, as amended by the Acts of March 3, 1933, and July 2, 1946 (Title 39, United States Code, Section 233) showing the ownership, management, and circulation of CORROSION, published monthly at Houston, Texas for October 1, 1952.

1. The names and addresses of the publisher, editor, managing editor, and business managers are: Publisher, National Association of Corrosion Engineers, 1961 M and M Bldg., Houston, Tex., Editor, Ivy M. Parker, P. O. Box 423, Bremen, Georgia, Managing editor, Norman E. Hamner, 1961 M and M Building, Houston, Tex., Business manager, A. B. Campbell, 1961 M and M Building, Houston, Tex.

2. The owner is: National Association of Corrosion Engineers, 1961 M and M Building, Houston, Tex.

3. The known bondholders, mortgagees and other security holders owning or holding 1 percent or more of total amount of bonds, mortgages, or other securities are: None. A. B. Campbell, Business Manager, sworn to and subscribed before me this 11th day of September, 1952, by Ann Graham. My commission expires June 1, 1953.

The favorable or unfavorable influence on corrosion by deposits on iron sheets depends on the type of force that binds the corrosive material to the deposits. If it is held physically, corrosion of the metal in water containing oxygen of carbon dioxide is greater than that of these same substances on deposit-free metals. If the corrosive material is retained chemically by the deposits present, however, corrosion is less than that of deposit-free metals.

### 3.5 Physical and Mechanical Effects

#### 3.5.3, 2.3.9

**Mechano-Chemistry of the Dispersion of Mercury in Liquids in an Ultrasonic Field—Effect in Cavitation Corrosion.** EVELYN C. MARBOE AND W. A. WEYL. *J. Applied Physics*, 21, No. 9, 937 (1950).

A note. The experiments were undertaken because of their relevance to cavitation corrosion; mercury was used to avoid work-hardening. Mercury could be dispersed readily in a liquid only if a strong polar bond was formed between the metal surface and the liquid. This was achieved: 1) by causing a monomolecular compound layer to be formed, e.g. the system mercury-paraffin-stearic acid; 2) use of liquids containing polar molecules, e.g. nitrobenzene; 3) introducing metallophilic ions such as  $Hg^{+}$  into solution. This ion could be formed in water by oxidation if dissolved air was present and it was found that distilled water would only allow dispersion of mercury if it contained dissolved air.—MA.

#### 3.5.8

**The Influence of Internal Stresses on Corrosion.** (In German.) E. FRANK. *Werkstoffe u. Korrosion*, 1, No. 10, 404-412 (1950) Oct.

A review of the literature dealing with: 1) origin and nature of internal stresses, 2) the effects of stresses on electrode potential and rate of corrosion, 3) the effects of the nature of the surfaces, 4) stress-corrosion in non-ferrous metals, 5) stress-corrosion in iron and steel, and 6) corrosion-fatigue. 202 references.—MA.

#### 3.5.8, 3.7.2, 6.4.2

**Investigations on the Properties and Stress-Corrosion of Aluminum Alloys of High Strength at Room Temperature—I-III.** B. W. MOTT AND J. THOMPSON. *Metal Treatment*, 14, No. 52, 227-238 (1947-48); 15, No. 53, 33-46; No. 54, 91-105 (1948).

Mott and Thompson investigated the tensile properties, age-hardening, resistance to fatigue, and stress-corrosion properties of 31 aluminum alloys in the composition range 1-5 percent magnesium and 3-12 percent zinc with 1 percent manganese. The effect of separate additions of 0.5 percent chromium, vanadium, and titanium to an alloy containing 3 percent magnesium and 7 percent zinc were also investigated. Results of stress-corrosion tests on R.R. 56 (D.T.D. 206), Duralumin (D.T.D. 356) and nickel-bearing R.R. 77 (D.T.D. 363) are also given. The results show that provided the percentage of magnesium or any other element (e.g. chromium) which may be added partially or wholly to replace manganese to keep within certain limits, alloys containing magnesium and zinc over a wide range of composition can be prepared which have satisfactory hot-working properties.



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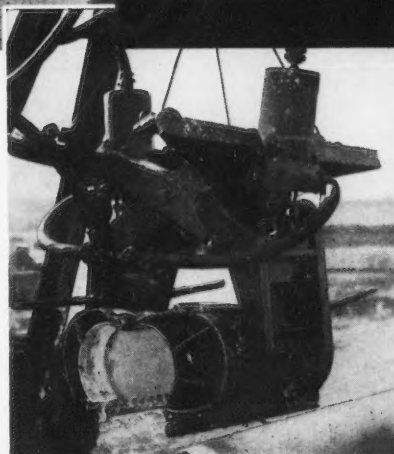
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and good mechanical properties in the fully heat-treated condition. The alloys can be forged or extruded into bar form, rolled to strip, or stamped in dies to intricate shapes. The hot-working properties were found to deteriorate seriously if the manganese (in the absence of chromium) exceeded 1.5 percent or if manganese + chromium exceeded 1 percent. In general, the proof stress and tensile strength increased and the ductility decreased with increasing magnesium and zinc content. The limits of composition for suitable alloys were reached when free MgZn<sub>2</sub> was present and the properties could not be improved by solution-treatment. The best results were obtained from alloys containing ~2 percent magnesium, 1 percent manganese, and from 10 to 11 percent zinc, typical tensile properties being ultimate tensile strength 40 tons/in.<sup>2</sup>, 0.1 percent proof stress 36.9 tons/in.<sup>2</sup>, elongation 5 percent. In general the alloys reached max. hardness on ageing at room temp. for 10 days and for alloys containing <9 percent zinc or <12 percent magnesium + zinc the best solution-treatment temp. was 495° C and the highest mechanical properties were obtained after ageing at 120° C for 16 hr. 1.4 percent Silicon was found to prevent age-hardening and it is stated that the silicon content in these alloys should be kept as low as possible. Fatigue tests on fully heat-treated extruded and forged materials gave higher values for the fatigue limit than are usually obtained on aluminum alloys, but chromium was found considerably to reduce the fatigue limit. An alloy containing magnesium 2, manganese 1, and zinc 11 percent gave a fatigue limit of 15.4 tons/in.<sup>2</sup> at 50 × 10<sup>6</sup> cycles. In general, the stress-corrosion properties were inferior to those of commercial aluminum alloys. The results indicate that ageing at 120° C does not cause any marked deterioration in the stress-corrosion resistance compared with that of material aged at other temp. Increase in zinc content caused some decrease in stress-corrosion resistance and chromium gave slight improvement in sheet material but a deterioration in extrusions. Microscopical examination of the alloys was carried out, and 25 microphotographs are given illustrating the structures obtained with these alloys. 40 references.—MA.

### 3.5.9, 3.5.8

**High Temperature Properties of Metals.** Book, 1951, 176 pp. Published by Am. Soc. Metals, Cleveland, Ohio.

Reproduces five lectures given in October, 1950. 1) "Creep of Metals" by E. R. Parker. 2) "Stress Rupture Testing" by N. J. Grant. 3) "High Temperature Fatigue Testing" by H. J. Grover. 4) "Methods of High Temperature Oxidation Testing and Evaluation of Observations" by C. Wagner. 5) "Some Experiences in Service (Power, Oil and Chemical Plants)" by J. J. B. Rutherford.

A detailed subject index is provided.—BNF.

### 3.5.9, 3.5.8, 3.2.2

**Properties of Metals at Elevated Temperatures.** G. V. SMITH. *Combustion*, 21, 65-67, 51-53 (1950) Apr., May.

Part I. Metal strength at elevated temperatures, working stresses that may be applied, characteristics of creep and the relation between stress and time for rupture. A typical design chart for a stainless steel.

Part II. Effects of non-constant stress and temperature, metallurgical variables, microstructural and surface changes and scaling and corrosion.—MR.

### 3.5.9, 5.4.2, 5.3.2

**High Temperature Refractories Discussed at Electrochemical Society Meeting.** *Steel*, 127, 78, 81-82 (1950) July 31.

Recent developments in refractories that will withstand 4000° F and higher; how chromium improves oxidation resistance of TiC (titanium carbide); protective coatings for molybdenum; vapor deposition of refractory coatings; improved palladium plating bath; new heat resistant protective coatings; and corrosion of composite plated coatings.—MR.

## 3.6 Electrochemical Effects

### 3.6.5, 6.3.4, 2.3.9

**The Anodic Behaviour of Metals.** Part VI. Cobalt. S. E. S. EL WAKKAD AND A. HICKLING. *Trans. Faraday Soc.*, 46, 820-824 (1950) Oct.

The initial build-up of anodic polarization at a cobalt anode over a wide range of conditions was investigated using the cathode-ray oscillograph. In alkaline solutions, three stages of oxidation were distinguished corresponding to the formation of CoO (cobaltous oxide), Co<sub>2</sub>O<sub>3</sub> (cobaltic oxide) and CoO<sub>2</sub> (cobalt dioxide). In passivation of a cobalt anode, the cobaltic oxide forms a protective layer, essentially 1 molecule thick, which is further oxidized to cobalt dioxide before oxygen evolution commences. 14 references.—MR.

### 3.6.5, 2.3.5, 5.2.1

**Studies on Galvanic Couples. I Measurement of Electromotive Force and Internal Resistance of Cells During Current Flow. II Some Potential-Current Relations in Galvanic Corrosion. III Polarization and Cathodic Protection.** H. D. HOLZER. *J. Electrochem. Soc.*, 97, Nos. 9, 12, pp. 271-282, 453-461 (1950) Sept., Dec.; *Corrosion*, 7, No. 2, 52-68 (1951) Feb.

I. A method of measuring the effective electromotive force and internal resistance of a galvanic cell during current flow is described. A dry cell was used for demonstration of the method. By introducing a reference electrode into a cell consisting of an iron electrode and a magnetite electrode in wet clay, it was possible to measure the potential and resistance between the reference and electrode and either the iron or magnetite during current flow. When the iron and magnetite were short-circuited, the potential and resistance between the reference electrode and the iron-magnetite couple were measured. By impressing current upon the galvanic couple from an auxiliary electrode, the potential-current characteristic of the couple was determined. It was shown that an iron plate in wet clay exhibits such a characteristic similar to that of the galvanic couple.

II. A graphic method of expressing the electrical characteristics of a galvanic couple was developed by the application of Kirchhoff's laws to a couple consisting of separate electrodes, each with an ammeter in series. When an external emf was impressed between the couple and a third electrode, also with an ammeter in series, a definite current pattern was established, depending on the relative directions and magnitudes of the impressed emf and the potential of

the couple. These relations were demonstrated with the iron-magnetite couple. Under the conditions used, a discontinuity in the potential-current relation was observed, when the couple was cathodically polarized over a sufficient range of current. This discontinuity of "break" coincided with reduction of current, circulating within the couple, to zero. The rise in potential at this point is the logical result to be expected from the hydrogen overvoltage.

III. The mechanism of polarization of a galvanic couple is developed. By means of separate elements in an electric network, the roles of electrode potentials and of resistive-potential differences are demonstrated in polarization and cathodic protection. Several methods of showing the relation between polarizing current and galvanic-couple potential are discussed. The electrical conditions which determine the distribution of current flowing to a galvanic couple are defined. The use of the term "current density" in cathodic protection should be carefully qualified; otherwise, it may be meaningless.

### 3.6.6, 5.1, 8.4.3

**Scale Deposit Prevention in Gas Lift Wells.** R. B. WALTER. *World Oil*, 130, 228, 230-231 (1950) June.

Use of down-the-hole assemblies of zinc and copper to set up galvanic action to prevent scaling on the pump, tubing, and other units of the flow string.—MR.

### 3.6.6, 1.3

**Illustrations of Galvanic Corrosion Given in Tabloid Primer.** A. B. LAUDERBAUGH. *Am. Gas J.*, 173, No. 6, 26-30 (1950).

A general description, with illustrations and drawings, is given of typical cases where galvanic corrosion may take place, together with details of precautions which should be applied.—MA.

### 3.6.6, 5.8.2, 6.2.5

**The Use of Chromates as Protection Against Galvanic Corrosion in the Systems Steel-Monel, Steel-Nickel, Steel-Inconel.** (In Italian.) L. CAVALLARO AND A. TANI. *Annali di Chimica*, 40, Nos. 8, 9, 10, 445-460 (1950) Aug.-Sept.-Oct.

Authors found that in sulfuric acid the action of chromates changed rapidly from inhibiting to stimulating the galvanic corrosion of steel when the pH exceeded a certain figure; results are correlated with previous work on the non-galvanic corrosion of various types of steels.—BNF.

### 3.6.8, 6.3.2

**Hydrogen Overvoltage on Individual Cadmium-Crystal Surfaces.** (In German.) E. ALBRECHT. *Z. Elektrochem.*, 55, 173-174 (1951) Mar.

Experimentally determined on mono-crystals of cadmium.—MR.

### 3.6.9, 7.7

**Isolation of Trolley Bus Negative Return To Prevent Stray Current Corrosion.** L. HORVATH (Ohio Bell Telephone Co.), H. E. NERHOOD (Ohio Edison Company), S. M. SEIDMAN (Ohio Bell Telephone Co.), and R. H. TRAVERS (Ohio Edison Co.) Paper, Eighth Annual Conference Nat'l. Assoc. Corrosion Engrs. Galveston, Texas, March 10-14, 1952. *Corrosion*, 8, No. 6, 205-211 (1952) June.

When a rail transportation system is converted to trolley bus operation, there is a tendency to continue the use of rails for negative return. In two Ohio cities—

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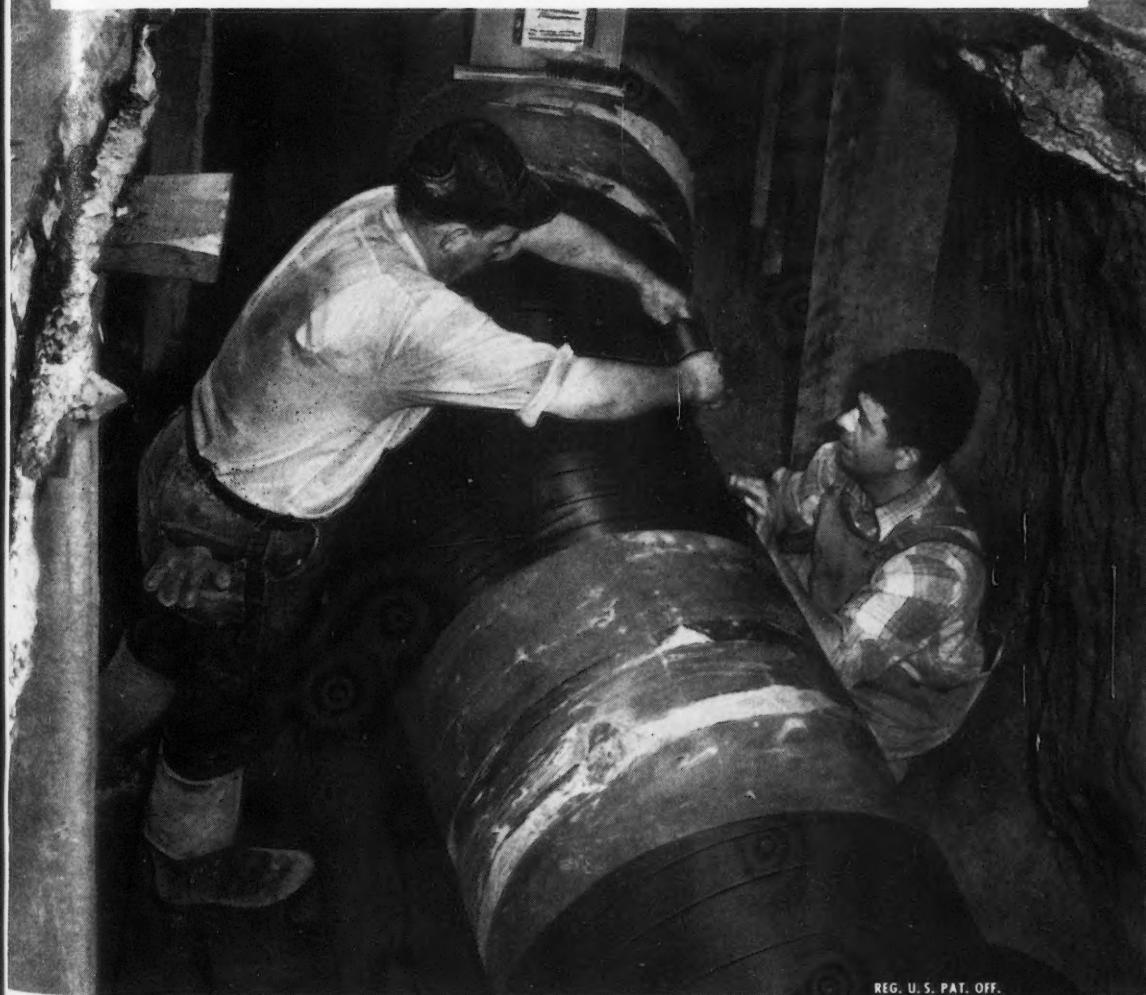
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Akron and Youngstown—where this was done, the resultant problems, corrosion and otherwise, finally led to abandonment of rail return and isolation of the negative return from ground.

The experience in these cities is reported from the standpoint of two utilities, Ohio Edison Company and The Ohio Bell Telephone Company, whose underground structures were involved. This paper compares corrosion conditions before and after the isolation. The problems which arose during the conversion and those which became apparent after the isolation are discussed.

## 4. CORROSIVE ENVIRONMENTS

### 4.2 Atmospheric

#### 4.2.1

**Vertical Distribution of Photographically Active Particles, Precipitated From Metals by Atmospheric Corrosion.** (In Russian.) I. L. ROIKH. *Doklady Akad. Nauk SSSR* (Reports of the Academy of Sciences of the USSR), new ser., **70**, 253-256 (1950) Jan. 11.

Claims that certain of the above effects were obtained with magnesium, aluminum and zinc. Quantitative data are given for variations of this effect with surface condition of the samples. No explanation is given for the peculiar phenomena reported.—MR.

#### 4.2.5, 8.9.5

**Some Aspects of Ship Bottom Corrosion.** PAUL FIEBEL, Superintendent of Development and Research Bethlehem Steel Company, Shipbuilding Division, Quincy, Mass. Presented, annual meeting of Soc. Naval Architects and Marine Engrs., New York, November 10, 1950. *Trans. Soc. Naval Architects and Marine Engrs.*, **58**, 608-657 (1950). *Corrosion*, **8**, No. 1 and 2, 29-48, 69-88 (1952) Jan. and Feb.

In the author's opinion the aspects of bottom corrosion of interest to the operator and shipbuilder can be summarized as follows:

Normally, hull steel corrodes slowly in sea water. It wastes away at a rate of less than 0.006 inch per year, which is equivalent to about  $\frac{1}{8}$  inch in twenty years. However, the problem facing the shipbuilder and operator is not one of general wasting away but rather one of localized pitting at higher rates of corrosion. Pitting is basically an electrochemical effect, the loss of metal occurring whenever electric currents flow from the metal to the sea water. Hull steel, due to its heterogeneous nature, has an inherent tendency toward pitting which generally can be tolerated. However, extraneous conditions may stimulate the flow of electric currents from the hull plating and cause extremely destructive pitting. The two most vigorous forms of hull corrosion due to pitting stem either from stray electric currents returning to a shore source or from galvanic currents. Especially destructive are those galvanic currents flowing from bare steel to mill scale.

Stray current corrosion is due usually to stray welding currents during outfitting and as such is essentially a problem of the shipbuilder. They can be controlled by placing the generators on the ship or with adequate ground returns from the ship to shore. It is extremely unlikely that electric currents generated within the ship when the vessel is at sea

would cause any corrosion on the outside of the underwater body, because there would be no inducement for the currents to stray from the hull plating.

Our industry has had to contend with mill scale corrosion for the last eighty years or more and with this background of experience it is surprising that we still encounter serious trouble from this source. The principal factors governing the occurrence of mill scale corrosion are: a) Rolling mill practice as it affects mill scale formation; b) Duration of weathering (plate yard storage and building period of the ship); c) Climate; d) The type of underwater paint selected; e) The maintenance of the underwater paint, including frequency of docking in the first year or two of service.

Mill scale corrosion occurs when the relationship of the foregoing factors is unfavorable and the sea water is able to reach relatively large areas of mill scale and small areas of bare steel.

The surest way of avoiding mill scale corrosion is to pickle or sandblast the underwater plating before the vessel is delivered. However, freedom from mill scale troubles can be obtained generally if the weathering is adequate, the paint is suited for application over mill scale, and the owner docks the vessel at three- and six-month intervals during the first year. Nonetheless, the presence of large areas of mill scale under the bottom paint can constitute a threat for a number of years, standing by to cause trouble if the bottom protection is allowed to deteriorate.

Vessels undergoing repairs involving renewals of bottom plates are especially susceptible to mill scale corrosion unless they receive the same care in painting and frequent docking which is accorded a new ship.

Zincs or Scandinavian irons do not necessarily protect the hull by sacrificing themselves (as in cathodic protection) but by shouldering most of the burden of corrosion caused by the propeller on the bare steel in its vicinity—and in effect concentrating the attack on replaceable "doubblers" which are not structural members.

Discussions by seventeen individuals are presented (*Corrosion*, **8**, No. 2, 69-82) followed by a reply by the author.

### 4.3 Chemicals Inorganic

#### 4.3.3, 6.1

**Resistance to Caustic Soda of Different Materials.** (In French.) A. GUITTON. *Metaux & Corrosion*, **25**, 84-86 (1950) Mar.

Corrosion resistances of alloy steels with a high content of nickel and molybdenum, Worthite, lead, enameled steel, rubber coatings, ferro-silicon, stainless steels and cement.—MR.

### 4.4 Chemicals Organic

#### 4.4.2, 5.4.5, 8.4.3

**Corrosion-Proof Coating for Oil Storage Tanks.** *Petroleo Interamericano*, **8**, No. 10, 40-41 (1950) Oct.

Use of Fiberglass Coromat pipe wrap to prevent corrosion in crude-oil storage tanks is described. About 120 squares of Coromat and 12-13 tons of coal-tar-blend enamel are used per tank. The 9 step procedure for treating tanks is described.

## 4.6 Water and Steam

### 4.6.4

**The Elements of Cooling Water Control.** L. WALTER. *Chem. Age* (London), **62**, 159-162 (1950) Jan. 28.

Automatic regulation of cooling water temperature and flow is described with diagrams of mixing valve or blender installations (i.e. for blending recirculated water with fresh water) and of self actuated thermostatic temperature control systems included.

#### 4.6.5, 7.6.4, 6.6.7

**Corrosion Problems in Ion Exchange Systems.** J. F. WILKES, Technical Director, Railroad Department, Dearborn Chemical Company, Chicago, Ill. *Corrosion*, **8**, No. 6, 1-2 (News Section), (1952) June.

Because deionization can produce water of extremely low mineral content, often at a fraction of distillation cost, it is used for high pressure boiler use and in numerous processes requiring mineral free water. The design and operation of de-ionizing plants presents many corrosion problems. In the deionizing process, mineral impurities are removed from water by chemical exchange reactions which are reversible. Because of the reversibility of the reaction, the resin beds can be reactivated by contact with regenerating solutions.

Because of the corrosivity of the extremely high purity water after going through the ion exchange system, rubber-lined steel exchanger tanks and rubber-lined, flanged pipe and fittings are used.

The acid regenerant system requires the use of stainless steel because of the difficulty in handling sulfuric acid. Deionized water is stored in wood-lined or steel tanks. Concrete lined tanks or concrete pipe are quickly eaten away by the high purity water. Surface coatings for concrete are being developed so that concrete can be utilized as a material of construction in the design of ion exchange systems.

## 5. PREVENTIVE MEASURES

### 5.2 Cathodic Protection

#### 5.2.1, 8.1.4, 8.9.3

**Cathodic Protection.** J. MULDER. *Water* (Holland), **34**, 241 (1950) Nov.

Discussion of theory and practice of cathodic pipe protection. Conclusion. Although this method of corrosion prevention is valuable, it is not a cure-all.—AWWA.

#### 5.2.1, 7.5.5, 8.1.4

**Experience with Cathodic Protection.** JAMES W. MCAMIS. *J. Am. Water Works Assoc.*, **42**, No. 6, 553-554 (1950) June.

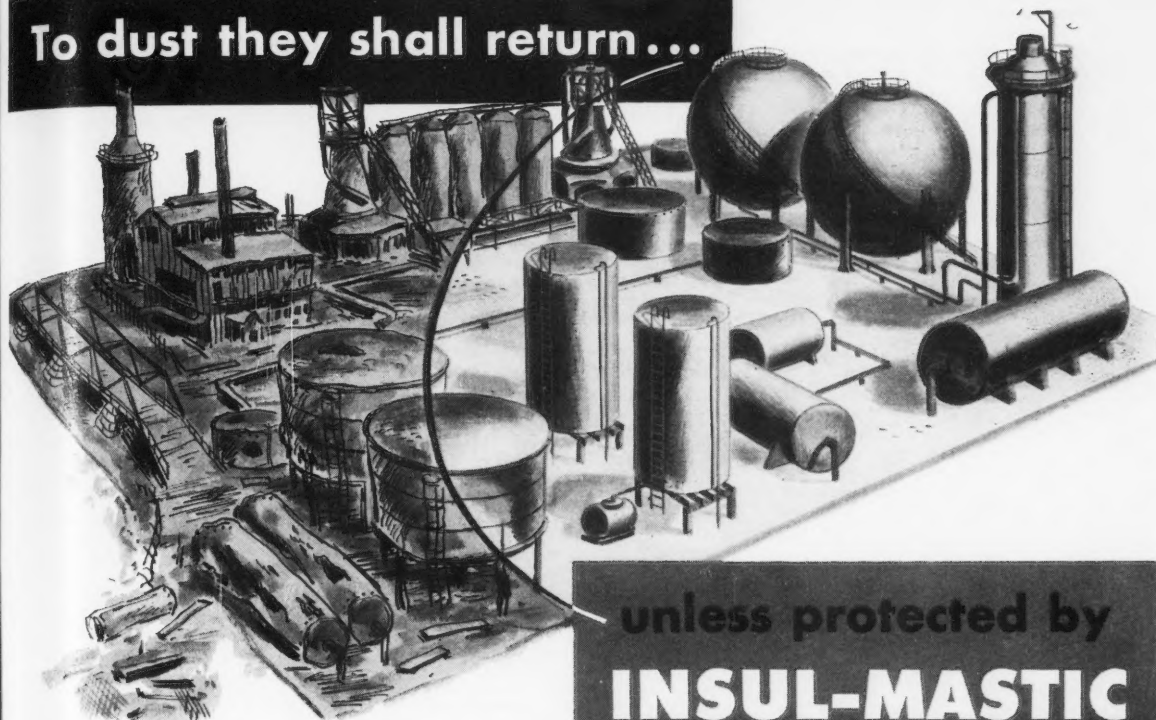
A brief note on the protection of municipal water tanks by painting and cathodic protection. Cathodic protection is believed to be more economical and easier in operation.—MA.

#### 5.2.2, 7.5.5

**Corrosion Mitigation Within Dehydrating Tanks.** ERNEST O. KARTINEN. *J. Petroleum Technol.*, **2** (1950) June; *Trans. Am. Inst. Mining Met. Engrs.*, **189**, 175-178 (1950).

Experiences with corrosion prevention in the water-exposed areas of dehydrating tanks and in some stock tanks. Results with various coatings were unsatisfactory.

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Having no by-products, we are free to choose the very best materials for long lasting corrosion prevention. Research has proven these to be Gilsonite, carefully chosen asbestos fibre and mica flake in large proportions. The result is a heavy coating ( $\frac{1}{16}$ " to  $\frac{1}{8}$ ") having considerable physical and chemical endurance.

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factory, but cathodic protection proved to be quite successful and economically feasible. Need for quantitative information concerning effects of various factors is indicated.—MR.

### 5.2.2

**Performance of Magnesium Galvanic Anodes in Underground Service.** OLIVER OSBORN AND H. A. ROBINSON. Paper, Sixth Annual Conference, Natl. Assoc. Corrosion Engrs., St. Louis, Mo., Apr. 4-7, 1950. *Corrosion*, 8, No. 4, 114-129 (1952) Apr.

The performance of magnesium galvanic anodes in underground service is reviewed in the light of the past five years of field testing in which upwards of 2500 anodes have been tested in some 40 different backfills and in various soils. The pertinent indices of galvanic anode performance are cited and the various factors influencing the performance of magnesium anodes are enumerated. A summary of the most outstanding findings relative to anode current efficiency, anode current output and stability of current flow is presented. The effects of such factors as anode composition and impurities, current density, time, backfill composition installation practice, soil characteristics, and weather on anode performance are discussed. The high purity Mg-6Al-3Zn alloy continues to provide the most consistently efficient performance. At current flows equivalent to ten year life, efficiencies corresponding to 500 ampere-hours per pound are usually realized except in high chloride soils where higher current densities are needed, to get comparable efficiencies. Impurities, notably nickel, iron, and copper must be held at low concentration levels in order to realize good anode efficiency. Pure commercial (cell) magnesium anodes occasionally exhibit high efficiencies but statistical experience shows their performance to be decidedly inferior to that of the high purity alloy. Field tests show the efficiency of the alloy anodes to increase with current density as previously indicated by laboratory data. Likewise, efficiency tends to increase with time for periods up to at least two years. With the exception of the magnesium sulfite and chromic acid backfills, the composition of the backfills used appears to have no very significant effect on anode efficiency.

Anode current output is essentially a function of soil resistivity and cathode potential although it is influenced to some extent by backfill composition and backfill installation practice. Statistical data indicates that backfills installed dry provide a higher level of current output than is obtained with wet (mud) backfills. Likewise, high conductivity backfills appear to provide significantly higher current flow.

Excellent maintenance of current flow has been observed for periods of three to five years on relatively large numbers of anodes. The most stable current flows are associated with deep installations and poor to fair soil drainage. Well drained installations on slopes or elevations tend to exhibit poor current maintenance. Depletion of soluble backfill ingredients (gypsum, sodium sulfate) is evident after three to four years of service but current flow is not adversely affected so long as the moisture supply is adequate. Bentonite rich backfills are highly moisture sensitive and tend to expand or shrink excessively with fluctuating moisture levels. If installed wet they sometimes shrink enough to adversely affect anode-to-earth resistance. Stability of

current flow is also affected by rainfall and temperature, the temperature effect being most pronounced where winters are coldest. Current flows are usually well maintained until the anode is 85-95 percent exhausted.

### 5.2.4, 7.2

**The Use of Cathodic Protection of Underground Metallic Piping.** A. J. MAURIN. *Tech. et appl. petrole*, 5, 1391-1401 (1950) Nov.

The nature of corrosion of underground metallic pipe is studied, and techniques of cathodic protection are considered for pipes carrying liquid and gaseous products as well as propane conduits. Economic aspects are briefly discussed.

## 5.3 Metallic Coatings

### 5.3.2, 1.7.1, 6.3.14

**Report of the Work of the Tin Research Institute 1949.** The Tin Research Institute (England), 1950, 16 pp.

Reports work on tin plate, hot-tinning, bearing metals, bronze, solder, electro-tinning, tin-zinc and tin-lead coatings, speculum plate, other tin-alloy coatings and on corrosion. Includes data on staff, committees, publications, exhibitions, etc.—MR.

### 5.3.2, 3.5.5

**Porosity of Electrodeposited Metals. VI. Effect of Surface Structure of the Base Metal on the Permeability and Corrodibility of Deposits.** N. THON, LING YANG AND DENIS KELEMEN. *Plating*, 37, 280-281+ (1950) Mar.

Deposits of the same metal, produced under otherwise identical conditions with unequal gas permeabilities of thin foils, generally corrode at unequal rates. Experimental data cover nickel and iron deposits on stainless steel and copper.—MR.

### 5.3.2, 1.6

**Thin Films and Surfaces.** WINIFRED LEWIS. Book, 120 pages. 1950. Chemical Publishing Co., 26 Court St., Brooklyn 2, N. Y. \$4.75.

Critical survey of the results of research on thin metallic films and surfaces with special reference to aluminum. Review of information concerning the structure, production and mechanical, optical, magnetic, electrical and chemical properties of thin metallic films and surfaces in general.—MR.

### 5.3.4, 2.3.7

**Research on Sprayed Metal Coatings.** (In German.) HANS VON HOFFE, WILHELM HOFMANN, AND GUNTHER SUCHAN. *Abhandl. Braunschweigischen Wiss. Gesellschaft*, 2, 175-184 (1950).

Steel was sprayed onto a steel shaft and the latter cut into cylindrical samples. By annealing the discs in hydrogen and in a vacuum between 900° and 1200° C, diffusion between the base steel and the coating was caused to take place. By this means, resistance to tension and shear was increased. Two steel discs were sprayed with brass and their brass sides placed in contact. These "double" samples were compressed in an Amsler machine and amount of compression measured. From these data, and similar ones for uncoated steel, elastic and plastic compression of the layer was determined. In a similar manner, cast iron, sintered iron and sprayed steel were studied. Graphs show comparative elastic and plastic properties.—BLR.

### 5.3.4, 6.2.3

**Recent Developments in Heat and Corrosion Resistant Coatings for Tools.** E. B. NEIL. *Tool Engr.*, 24, No. 3, 34-36 (1950) Mar.

Surfaces of dies and other tools subjected to wear and abrasion are being hard surfaced either by welding the hard materials to the surface, or by electrodeposition of the material. Newest of the hard metal coatings, applicable by arc-welding procedures is a chromium-carbide powder. This is mixed with water to form a paste which adheres to flat and curved surfaces. Its use is recommended where application by means of hard surfacing electrodes is undesirable, such as thin deposits, on thin work, and for use with a.c. welders. Primary undercoatings of nickel and other metals may prove more satisfactory than direct plating of the hard metal on the steel surface of the tool. The electrodeposition of wolfram (tungsten) and of nickel-cobalt alloys on steel and of chromium on aluminum (where both low weight of the tool, wear and corrosion resistance are advantageous) are new possibilities. In chrome plating, pitting of the chrome surface can be avoided only by providing a sufficiently thick deposit of copper, nickel or other more highly resistant metal under it. The corrosion resistant characteristics of the base metal influence the thickness of the undercoating. Another development is the plating of plastics on metal to protect the tool from wear and corrosion. Nor should the depositing of white brass, black molybdenum, black nickel, lead-tin, brass, bronze, silver, indium and other rare metals be overlooked. Each has its characteristics in resisting corrosion and giving a decorative finish. Ceramic and organic coatings are also discussed.—INCO.

### 5.3.4

**Mirrors of Motordom.** A. H. ALLEN. *Steel*, 129, No. 6, 57-58 (1951) Aug. 6.

"White brass" plating, developed by DuPont, involves a cyanide type of plating solution with molybdic acid additives, brightening agents, plus anodes of 80 percent zinc and 20 percent copper. It can be applied to zinc, brass, or steel with conventional equipment and with a plating cycle slightly slower than bright zinc. The maximum thickness is 0.0003-0.0004 inch, which is followed immediately by a chromium flash since white brass will tarnish unless thoroughly rinsed in cold water and dried. The new plating has withstood 48 hour salt spray test, against 16 hours now specified for copper-nickel-chromium plates. The control of the plating is critical, but the parts are mirror bright and require no buffing.—INCO.

### 5.3.4, 6.4.2

**Electroplated Coatings on Aluminum Alloys.** J. H. JAMES. U. S. Naval Air Material Center, Philadelphia. Report AML NAM AE 411030, Part III. September 1950.

The corrosion resistance of 3S-O, Alc 24S-T4, and 75S-T6 aluminum alloys plated with nickel or chromium was evaluated by means of salt spray, humidity cabinet and atmospheric exposure tests. Nickel coatings are in general more resistant than chromium coatings, particularly in atmospheric exposure. When chromium plating is to be used as hard surfacing material for aluminum alloys, its application should be preceded by that of nickel plating at least 0.002



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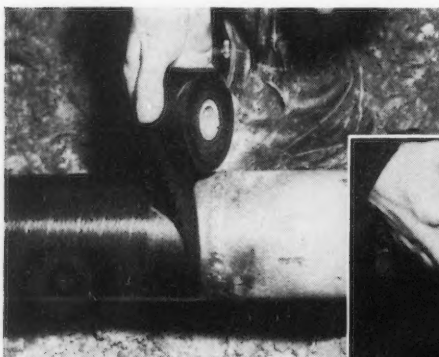
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inch thick for optimum corrosion resistance.

Within the ranges studied, the protection against atmospheric exposure afforded by nickel coatings is at least equal to that conferred by chromium coatings 4 times as thick. A nickel thickness of 0.001 inch per year of atmospheric exposure is required for 3S-O alloys; somewhat greater thicknesses are necessary for Ale 24S-T4.

When exposed to 20 percent sodium chloride spray at 95° F, a chromium coating as thin as 0.0001 inch over nickel-coated 3S-O and Ale 24S-T4 about doubles the corrosion resistance of a given thickness of nickel plate alone.

Humidity cabinet exposure (100 percent RH, 120° F) for 16 months did not appreciably affect nickel plated 3S-O and Ale 24S-T4 sheets or nickel- or chromium-plated 75S-T6 samples.

### 5.3.4

**Current Technical Problems in Metal-Spraying.** PUSCHEL, *Metallüberfläche* (A), 4, No. 9, 141-143 (1950).

Some of the outstanding problems associated with the spraying of metals are postulated and discussed. They are concerned with the relative technical and economic merits of: 1) the various types of equipment; a) fixed or portable, b) compressed air or electric motor feed of the wire, c) constant pressure or injector methods of supplying the gaseous fuel, and d) the various types of fuel; 2) the diameter of the wire; 3) the use of neutral or reducing atmospheres to prevent oxidation of the metal particles, in relation to their effects on the hardness and porosity—and, consequently the adherence and the corrosion-resistance—of the coatings produced; 4) spraying versus galvanizing and electrodeposition; and 5) the spraying of steel on to light metals.—MA.

### 5.3.4, 5.9.2

**Washing After Pickling in Wet Galvanizing.** (Le lavage apres decapage dans la galvanisation avec couverte.) A. GORDET, *Metallurgie* (France), 82, No. 10, 789-793 (1950) Oct. 3000 Words.

The author strongly emphasizes the advantages of rinsing after pickling articles which are to be dipped through a flux blanket without prefluxing. He claims that works trials lasting 5 months have shown, that dross formation is reduced by 75 percent and that there is an appreciable saving in costs in spite of the additional stage in the galvanizing process. To avoid the danger of washed work rusting while waiting to be galvanized, the author recommends a dip in a neutral cold flux solution, after which work may be left for several hours without deterioration.—ZDA.

### 5.3.4, 5.3.2

**Chemical-Dip Coatings.** J. F. STIRLING, *Practical Engr.*, 22, No. 559, 382-383 (1950).

A short review of some well-known methods of producing protective coatings on aluminum, zinc and magnesium and their alloys on tinplate.—MA.

### 5.3.4, 5.3.2, 1.3

**Electroplating and Metal Finishing Developments in Germany: 1940-1950.** R. SPRINGER, *Proc. Am. Electroplaters Soc.*, 37th Ann. Conv., 1950, 91-103.

Literature review (100 references, including patents) covering cleaning and pickling, nickel, chromium, zinc, silver.

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gold and iron plating; anodizing of aluminum; protective coatings on magnesium; phosphating, etc.—BNF.

### 5.3.4, 8.3.5

**Tinplate or Blackplate for Food Cans.** G. SCHRÖDER. *Verpackungswirtschaft*, No. 1, 18 (1950); *Fette u. Seifen*, 52, No. 9, 570 (1950).

Requirements for food can varnishes are discussed. Blackplate food cans coated with lacquers based on phenolic resins are highly acid-resistant and may be used for fruit preserving. Even tinplate cans must be lacquered for certain kinds of foodstuff.—RPI.

### 5.3.4, 6.3.19

**The Metallurgy of Metal Spraying (Especially Zinc).** K. KREKELER. *Metalloberfläche* (A), 4, No. 10, 151-153 (1950).

After a brief outline of the oxy-acetylene wire process, Krekeler discusses the properties—adhesion, flexibility, and corrosion resistance—of the coatings obtained in relation to: 1) the nature of the basis metal, 2) the particle size of the sand used for sand-blasting preparatory to spraying, 3) the thickness of the coating, and 4) the operating conditions of the pistol, namely a) wire diameter and rate of feed, and b) the nature of the flame, whether oxidizing, neutral, or reducing. Photomicrographs and graphs illustrate the effects of these variables in relation to the properties of sprayed zinc coatings.

## 5.4 Non-Metallic Coatings and Paints

### 5.4.2, 5.4.5, 8.9.3

**Fiberglass Outer Wrap Provides Protection.** *Pipe Line News*, 22, No. 11, 34 (1950) Nov.

Fiberglass Pipeline Outer Wrap has been announced by Owens-Corning Fiberglass Corp. It is a bituminous composition reinforced with glass fiber whose characteristics are listed as: 1) protection for enamel coating, 2) prevents damage during lowering operation, 3) safeguards against backfill damage, 4) equalizes coating thickness around the pipe, 5) lessens soil stresses on coating enamels, 6) keys in with the enamel to form a unified protection for pipe lines, and 7) it has a low moisture absorption.

### 5.4.2

**Using Ceramic Coating For Specialized Applications.** WALTER RUDOLPH. *Finish*, 7, 19-21 (1950) Apr.

High-temperature ceramic coatings prove useful for applications varying from pump cylinders to components for B-36 bombers and jet engines.—MR.

### 5.4.3, 8.9.3

**Inspection of Welds in Gas Pipe Lines by Means of Gamma Radiation From Radium.** (In Russian.) R. B. POLYAKOVA. *Avtogennoe Delo* (Acetylene welding), 21, 18-19 (1950) July.

Method and apparatus developed for inspection under field conditions.—MR.

### 5.4.5, 5.8.4

**Protection of Iron Against Rust.** C. SALVI. *Pittura e vernici*, 6, No. 12, 43-44 (1950).

Directions are given for the degreasing, descaling and painting of old and new iron work and the relative values of  $Pb_3O_4$  (red lead oxide) and zinc chromate as rust inhibitive pigments are briefly discussed.—RPI.

### 5.4.5, 5.4.8

**Painting of Steel Mill Buildings and Equipment.** W. W. SCHERER. *Iron Steel Engr.*, 27, 60-63 (1950); *J. Iron Steel Inst.*, 167, Pt. 4, 458 (1951).

The importance of preparing surfaces before painting, of applying coats under proper conditions, of providing an adequate thickness of paint and of selecting the correct type of paint is discussed. The properties of primers and paints are examined in relation to service and maintenance requirements. The relative merits of oil paints, varnishes, enamels, pitch paints, lacquers and water paints are considered. The influence of colors on the worker's performance and attitude is discussed. The importance of pleasant surroundings by planned color schemes can no longer be neglected. General rules for relieving eyestrain and improving concentration in order to contribute to the general works efficiency and to produce the best psychological effects on the workers are indicated.—RPI.

### 5.4.5, 5.9.3

**Plastic-Coating with Polyethylene.** M. DUVIVIER AND H. F. HEDDE. *Ind. Plastiques Mod.*, 2, No. 7, 16-17 (1950).

Pure polyethylene can be flame-sprayed to give a coating resistant to chemicals, electrically insulating and useful in a variety of applications, e.g., to coat light-metal parts of aircraft electrical accumulators, or to coat electroplating baths. Three sizes of pistol are used: laboratory-scale; small-scale, covering about 1-1.5 sq. m./hr. to a depth of 1 mm.; and large-scale, covering about 6 sq. m./hr. to a depth of 1 mm. The surface to be coated is first cleaned and freed from grease, preferably by light sand-blasting if metallic. The spraying is best done at right angles to the surface from about 15-20 cm. A coating of lead 3 mm. thick can be replaced by a 1 mm. coating of polyethylene costing less than one-sixth of the price of the lead coating.—RPI.

### 5.4.5

**Yellowing.** R. J. LEDWITH and Others. *Paint Technol.*, 15, No. 178, 434-435 (1950).

An OCCA Brains Trust discussion held on the yellowing of varnish and resin films on exposure to moderate heat or light is reported in full (cf. *Review*, 1951, 151). It is considered that there is no direct relationship between the two ageing conditions since, e.g., phenolic resins yellow on heating but are bleached in light. The type of radiation, e.g., whether carbon arc or mercury arc is used, is important, since some resins yellow under one and not under the other.—RPI.

### 5.4.5, 2.3.9

**Size, Shape and Weight of Molecules of Vehicles Used in Research Programme.** J. S. LONG AND W. R. LAWTON. *Official Digest Federation Paint & Varnish Production Clubs*, No. 311, 1006-1011 (1950).

Molecular weights and molecular cross-sectional areas, determined with a conventional Langmuir balance, are given for the media used by the CDIC Club (*Review*, 1951, 156).—RPI.

### 5.4.5, 2.2.5

**Qualification Testing of Organic Finishing Materials: Preliminary Results of Florida Exposure Tests of New Lacquer**

**and Primer Formulations.** R. A. MACHLOWITZ. U. S. Naval Air Material Center, Report AML NAM AE 424338(7), 1950, Pt. 1, 14 pp.

Results of a six-month exposure test on 4 air-drying primers and 12 lacquers are given. The lacquers, which varied in plasticizer/alkyd/N.C. ratios, gave equal performances. Primers containing up to 5 percent dispersion resin formed more flexible systems than those containing 10 percent. Embrittlement was increased by replacing the unmodified alkyd in a primer by an equivalent weight of a copal-modified alkyd. Systems based on a second coat of primer retained greater flexibility.—RPI.

### 5.4.5

**Improved Corrosion Inhibiting Pigments.** M. L. KASTENS AND M. J. PRIGOTSKY. *Die Castings*, 8, No. 2, 44-49 (1950) Feb.

Barium potassium chromate pigment is used in the formulation of improved primers for the light metal alloys, aluminum and magnesium. The protective effect is better on aluminum and magnesium than on the ferrous metals, 20-25 percent more chromate ion per unit weight is added to enhance the anticorrosive action of the paint. Although it is a true double salt it hydrolyzes to release only potassium chromate and the barium chromate remains as a coating. The metals barium and potassium do not promote polymerization of the vehicle oils catalytically, and the new pigment has a low tinting strength so it may be included in finish coats.—INCO.

### 5.4.5, 2.3.6

**Macro and Micro Pictures of Paint Failures.** I-II. R. KLOSE. *Ind.-Lack-Betrieb*, 18, No. 9, 161-165; No. 10, 184-187 (1950).

The investigation of paint failures in order to assess their probable cause is discussed. The article is illustrated by micrographs of paint film sections and paint surfaces.—RPI.

### 5.4.5

**Paint Industry in Japan.** S. IWAI. *Paint Varnish Production Mgr.*, 30, No. 11, 51-53 (1950).

The development of the Japanese paint and varnish industry from the early use of anti-corrosive paints for ships to the present day is described. Statistics are given of the relative production of the various types of product.—RPI.

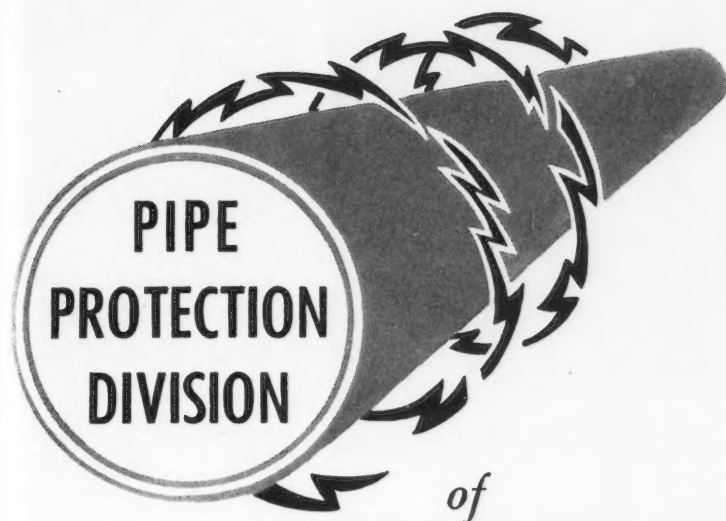
### 5.4.5

**Danger of Rusting With Acid-Hardening Lacquers.** *Ind.-Lack-Betrieb*, 18, No. 11, 207-208 (1950).

Immediately before application, acid-hardening lacquers contain about 1-2 percent free hydrochloric acid or sulfuric acid, and it is some days before the hardened film reaches neutrality. Although wooden or metal parts are suitable for assembly within 24 hours of coating with such lacquers, the residual acidity in the film may rust bare iron screws and corrode brass used in assembly. A simple test is described which showed that the acidity of an acid-hardening lacquer film five days old had diminished to negligible proportions.—RPI.

### 5.4.5

**Paints Based on Linseed Oil and on Bitumens.** K. MEIER. *Bitumen, Tere, Asphalt, Peche*, 1, No. 8, 219-221 (1950); *Peintures pigments vernis*, 27, No. 1, 40 (1950).



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The efficiencies of coatings based on linseed oil and on bitumens in preventing corrosion are compared. Bitumen emulsion paints show particularly good resistance.—RPI.

**5.4.5, 2.5, 1.7.1**

**Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer and Related Products.** D.609. 50T. *ASTM Preprint No. 80*, pp. 48-50 (1950).

The proposed revised tentative standard prescribes the type of steel panel to be used and the procedure to be followed in its preparation for testing paints, etc. Provision is made for panels to be used without chemical treatment and after phosphating.—ZDA.

**5.4.5, 6.4.2, 4.2.5**

**Paint Protection for Aluminum in Marine Atmospheres.** *Aluminum Courier*, No. 14, 10 (1950); *J. Brit. Shipbuilding Res. Assoc.*, 6, No. 4, 246 (1951).

Several proprietary methods for the chemical pretreatment of aluminum are described. Wash primers promote adhesion of paint and a zinc chromate pigmented primer is recommended. Primers containing lead oxide, lead chromate, graphite, or bronze powder should be avoided. Normal marine finishing coats may be used, but anti-fouling compositions specially for use with aluminum have been developed.—RPI.

**5.4.5, 6.6.7, 4.3.6**

**Chlorinated Rubber Corrosion-Resistant Coatings.** F. K. SHANKWEILER, G. N. BRUXELLES AND R. E. WHITNEY, Hercules Powder Company, Wilmington, Del. Paper, Seventh Annual Conference, Natl. Assoc. Corrosion Engrs., New York, N. Y., March 13-16, 1951. *Corrosion*, 8, No. 4, 130-139 (1952) Apr.

Industrial experience during the past fifteen years has shown that chlorinated rubber is an outstanding film-forming material from which corrosion-resistant coatings may be formulated.

Correct formulation has a marked beneficial effect on resistance of chlorinated rubber systems to corrosive solutions or atmospheres. For maximum chemical resistance the types of modifiers for chlorinated rubber are restricted to chemically inert resins and plasticizers which must be formulated properly to give optimum film properties and satisfactory weathering resistance.

If proper precautions in selecting the modifying components are not considered, corrosion resistance of chlorinated rubber coatings may be seriously impaired. For example, alkali resistance rapidly diminishes when the vehicle is modified with saponifiable resins or plasticizers. Similar deficiencies develop if components are incorporated which are not themselves unaffected by acids or salts.

Pigmented coatings in which the film consists of 50-60 percent chlorinated rubber, 15-20 percent Aroclor 5400, Chlorowax 70 or hard coumarone resins, etc., and 25-30 percent Clorafin 42-S, Aroclor 1254 or Cumar P-10, etc., are shown to provide highly effective protection to corrosion. Panels with such coatings tested in the salt fog (ASTM B-117-44T, 20 percent sodium chloride, 100° F) and in tap water immersion (100° F) in comparison with many other types of coatings illustrate their value when exposed to salt corrosion or water.

Because best performance cannot be obtained without suitable primers, a

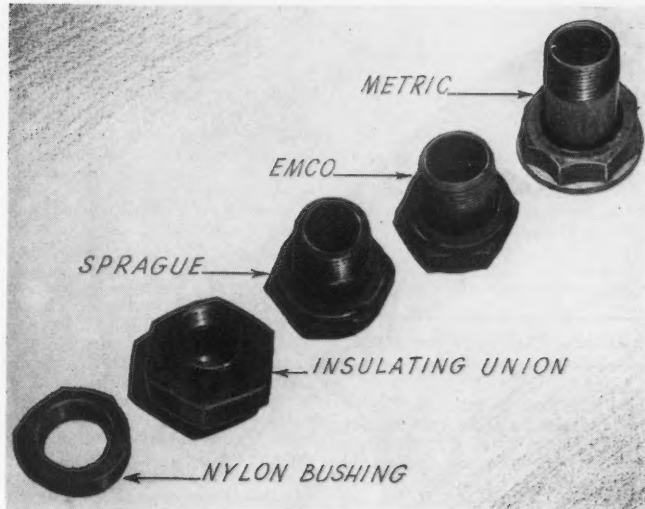


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comparison of chlorinated rubber and other types of primers was undertaken in these tests. It is shown that pigmentation of chlorinated rubber primers is equally as important as that previously established with other vehicles. When properly formulated with corrosion-resistant pigments, chlorinated rubber primers together with chlorinated rubber topcoats afford a high degree of protection.

Numerous case histories of commercial use in varied maintenance problems where chemical resistance is essential establish the economical merit of such coatings. Among the fields discussed are acid plants, paper mills, water works, industrial machinery, sewage disposal plants and concrete construction protected by chlorinated rubber.

One feature of chlorinated rubber coatings has led to their acceptance for industrial maintenance. They are applicable by brushing or spraying to surfaces which cannot be physically prepared by ideal procedures. Chlorinated rubber, like most coatings, gives best results over rust-free, clean metal to which efficient primers are applied. The outstanding feature of chlorinated rubber, however, is that it affords reasonably good protection over many types of undercoats or over poorly prepared surfaces.

## 5.8 Inhibitors and Passivators

### 5.8.2, 2.3.3, 2.2.7

**Further Field Test Results on Use of Corrosion Inhibitors and Bactericides for Secondary Flood Waters.** E. T. HECK AND J. K. BARTON (Quaker State Oil Refg. Corp.) and W. E. HOWELL (Hercules Powder Co.). Paper, A.P.I. Eastern District Meeting, Pittsburgh, Apr. 29, 1949. *Producer's Monthly*, 13, No. 7, 27-34 (1949) May; *Oil Gas J.*, 48, No. 18, 83- (1949) Sept. 8.

Three corrosion inhibitors, Rosin Amine D acetate, Pur-O-San and Arquad S, were evaluated on six different locations in the Bradford field. The method used was based on the previously reported procedure (J. N. Breston, *Bull. III*, 10(d) (1948) Jan. 15) in which corrosion protection was determined by weight loss data on steel coupons, bacteria count, and visual examination of water used for flooding. Good control was obtained when the inhibitors were used in the range of one to ten p.p.m. Depending on the type of water used for flooding, good to excellent protection was achieved. Leases on which flood waters contained hydrogen sulfide and carbon dioxide were found to be well adapted to the use of amine inhibitors. Slightly less effective results were reported on leases flooded with water containing dissolved oxygen. However, the results on both of these flood waters were considered satisfactory and an expansion of this treatment is now underway. Negative results were obtained on one lease in which the pH of the flood water was adjusted to 4.0 and 5.0. Although no attempt was made to measure turbidity quantitatively, it was evident that a marked decrease was obtained with all three inhibitors tested. The effect was most striking on the Fisher and Demps leases where the water contained hydrogen sulfide. Corrosion and bacteria count data are included.

### 5.8.2, 4.2.7

**Where Rust Doth Corrupt.** *Civil Engr. Corps Bull.*, 4, No. 43, 153-156, illus. (1950) June.

Large-scale dip-application of a commercial rust arrester, Spec. MIL-C-10036(ORD), permits preservation and storage of huge amounts of war surplus metal construction materials in the tropical climate of the Pacific islands at less than 4 percent of replacement cost. The method appears adaptable to various other types of metal items in storage or transit and to the maintenance of metal parts in general.

The crates or bundles of steel building materials are carried from storage by a fork-lift truck to the processing area, where loose scales and dirt are removed with an airgun. The crates are then lifted with a crane and submerged 3-10 minutes in a dip tank installed below the ground level and containing a solution of the rust arrester. The solution penetrates the rusted material to the base by capillary action, driving out all water, and incorporating the rust as a sort of pigment while forming a thin, flexible, transparent film which does not crack under daily temperature fluctuations and which is readily painted. The treated bundles are raised to permit drainage of excess fluid into the tank, and then swung over to a drain rack and left for 15 minutes. Continuous swabbing of drain-rack decks prevents solidification of the preservative and permits maximum fluid recovery. After draining, a fork lift carries the bundles to temporary horizontal storage on level dunnage; a firm, uniform coating forms within one week.

For permanent storage, the material is raised well above ground using heavy dunnage blocks, and stacked in a sloping position for efficient moisture drainage. Ends and tops of stacks are sprayed with an extra coat of preservative to cover any scars received during handling and final placement in storage. The entire treatment costs less than \$10 per ton material as compared with an estimated replacement cost of over \$250 per ton.—PDA.

### 5.8.2, 5.8.4, 5.6.2, 2.3.2

**Dicyclohexylammonium Nitrite, A Volatile Inhibitor for Corrosion Preventive Packaging.** A. WACHTER AND T. SKEI (Shell Development Company, Emeryville, Calif.) AND N. STILLMAN (Western Foil Converters, Inc., Berkeley, Calif.). Paper presented at the Seventh Annual Conf. Natl. Assoc. Corrosion Engrs., New York, N. Y., March 13-16, 1951. *Corrosion*, 7, No. 9, 284-294 (1951) Sept.

Dicyclohexylammonium nitrite is a powerful inhibitor for corrosion of steel by fresh water. Since this nitrite salt is slightly volatile, it has commercial application in a new method for preventing corrosion of packaged steel articles by the moisture that enters or is present within a package. It functions by vaporization and transport of its vapor to the metal surface where it condenses or dissolves in condensing moisture. The duration of protection is dependent upon the quantity of inhibitor present and the rate of loss as vapor and by decomposition. The compound has properties of chemical stability, compatibility, volatility, vapor transport, diffusion, solubility, and corrosion inhibition that are important to its functions as an inhibitor in packages. The absence of undesirable effects with nonmetallic materials, nonferrous metals, bimetallic couples and in handling by people are also of practical importance.

A large number and variety of tests in the laboratory and in outdoor exposure racks have given information on

the performance of the inhibitor under many different conditions. A dynamic corrosion test has been developed that is useful for rapid evaluation of volatile corrosion inhibitors for packaging applications.

The inhibitor is most conveniently and effectively employed in the form of a coating on wrapping papers or on paper-board containers. In order to obtain good results it must be used properly. It is not a panacea.

### 5.8.2, 5.9.2, 2.6

**Investigation of Some Pickling Inhibitors.** S. A. BALEZIN AND S. K. NOVIKOV. *J. Applied Chem. (USSR)* (*Zhurnal Prikladnoi Khimii*), 24, No. 3, 283-288 (1951).

Investigational arrangement and procedure. Study of suitability of formaldehyde as pickling inhibitor over a wide range of concentrations. Abnormalities observed. Hexamethylene tetramine as inhibitor in sulfuric acid pickling of steel. Results of experiments on action of thiodiglycol (broad range of concentrations) in sulfuric acid vs. hydrochloric acid. Mechanism of reactions involved. Influence of hexamethylene tetramine and thiodiglycol on temperature coefficient of rate of dissolution of steel in sulfuric acid. Translation available Henry Bratcher, P. O. Box 157, Altadena, Calif.

### 5.8.2, 6.6.11

**Chromate as a Corrosion Inhibitor in Wood Preservatives.** H. HADERT. *Werkstoffe u. Korrosion*, 2, No. 2, 49-51 (1951).

The author disagrees with previous reports on the effectiveness of chromate, maintaining that the chromate may react chemically with the wood preservative or the wood itself, possibly forming acid corrosive products.—RPI.

### 5.8.3

**Physical Chemical Aspects of Corrosion Inhibition.** NORMAN HACKERMAN, Department of Chemistry, University of Texas, Austin. Paper, Seventh Annual Conference, Natl. Assoc. Corrosion Engrs., New York, March 13-16, 1951. *Corrosion*, 8, No. 4, 143-149 (1952) Apr.

Some of the basic physical and chemical phenomena which can occur during the inhibition of the corrosion of metals by liquid environments are discussed. The way in which each of these may be involved and their relative importance in the over-all process is considered. Several recent researches in this field are examined to point up the variety of physical chemical approaches used.

### 5.8.3, 5.8.4

**Contribution to the Study of Pickling Inhibitors.** (In French.) G. BATA, L. SCHEEPERS, AND L. BOUSMANNE. *Rev. Met.*, 48, 105-114; disc., 114-115 (1951) Feb.

Theoretical considerations on corrosion inhibitors show that a relationship exists between molecular structure and inhibitory action of organic compounds on iron, aluminum and nickel. The same inhibitors behaved abnormally on zinc, alcohols, aldehydes, ketones, amines, phenols, halogenated hydrocarbons and CS, were investigated.—BLR.

### 5.8.3, 5.8.4, 1.3

**Chemical Treatment to Mitigate Corrosion. A Review.** J. G. JEWELL. Presented at a meeting of Pittsburgh Section, Natl. Assoc. Corrosion Engrs., April 5

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1951. *Corrosion*, 8, No. 3, 100-108 (1952) Mar.

A review of surface active and ionic type corrosion inhibitors, methods of neutralization and buffering, removal of corrosive agents, self-repairing coatings and bactericides are discussed. Anodic and cathodic agents are considered at length and the various factors incident to the use of these types of inhibitors described.

Industrial applications of anodic and cathodic agents are given in detail, percentages of some agents customarily used for specific services are listed. Organic inhibitors, adsorption inhibitors, pickling inhibitors, oil-wetting agents and special applications of anti-corrosion additives are considered.—BLR.

#### 5.8.4, 4.4.3

**Aqueous Nonflammable Hydraulic Fluids.** J. E. BROPHY, V. G. FITZSIMMONS, J. G. O'REAR, T. R. PRICE, AND W. A. ZISMAN. *Ind. and Eng. Chem.*, 43, 884-896 (1951) Apr.

Describes research which led to development of nonflammable hydraulic fluids designated as Hydrolubes, which are defined as polymer-thickened, corrosion-inhibited, aqueous fluids having one or more glycols as major constituents. The most suitable of those investigated were derivatives of the polyalkylene glycols. The large number of metals occurring in aircraft hydraulic systems posed a difficult problem in corrosion inhibition. However, suitable liquid-phase and vapor-phase inhibitors were found. Wear-reducing additives were developed of the type forming hydrophobic films on steel. 18 references.—BLR.

#### 5.8.4, 5.8.2, 7.1

**Motor-Oil Additives.** J. C. GENIESSE AND W. SCHREIBER. *Atlantic Refining Co. Oil Gas J.*, 49, No. 47, 302-305, 307 (1951) Mar. 29.

Advances in engine design have resulted in widespread use of additives both inhibitor and detergent, in motor oils. Use of alloy-type bearings such as copper-lead and cadmium-silver has made it necessary to incorporate chemicals of the oxidation-corrosion inhibitor type into the oil in order to prevent corrosion under high-temperature operation. The detergent-dispersant type of additive is used to take care of ring and piston deposits. Considerable progress has been made by addition of rust preventives, such as metallic soaps and esters. With the development of the radioactive tracer technique for measuring engine wear, superior additives should be the result. 5 references.—INCO.

#### 5.8.4, 5.9.2

**Derusting Equipment in the Field—The Use of Iodine as a Pickling Inhibitor.** K. J. HAGER AND M. ROSENTHAL. *Ordinance*, 35, No. 185, 479-480 (1951) Apr.

Acid pickling is discussed as commonest method of rust removal, where geometric shape is of no consideration and where the hollow interior of rusted equipment must be reached. Iodine has been proved to be an inhibitor when used in crystal or tinctures with any usually obtainable acid, ranging from hydrochloric acid through to acetic acid. Discussion includes importance of oiling and lessened danger of hydrogen gas embrittlement of steel during field pickling.—INCO.

#### 5.8.4, 8.4.3

**Corrosion Mitigation in the North McCallum Field. Parts I, II.** C. C. FRYE, W. L. GIEZENTANNER AND F. M. CLEMENT. *Continental Oil Co. World Oil*, 132, 138-140, 142 (1951) Mar.; 214 (1951) Apr.

Part I. Severe corrosion of tubing and casing in the distillate wells of the North McCallum field, Colorado, led to operating tests with various inhibitors including manganese naphthenate, ammonium hydroxide and three proprietary compounds. The methods of conducting the tests are described.

Part II. Reports on results obtained through the use of commercial inhibitors, and compares results so obtained against bare tubing and strings coated with plastics. Their findings indicate that liquid inhibitors are effective and that plastic-coated strings are of economic value in reducing the amount of inhibitor required.

### 5.9 Surface Treatment

#### 5.9.2

**Formulation of Paint Removers.** C. T. & F. D. SNELL. *Chem. Industries*, 64, 414-420 (1949); *Paint Notes*, 5, No. 7, 210 (1950).

New developments in the formulation of paint removers are discussed. Modern paint removers usually contain a number of ingredients to provide an all-around product which will meet as many of the following objectives as possible: 1) rapidity of action, 2) low volatility, 3) proper viscosity, 4) non-toxicity, 5) low inflammability (for some types), 6) clean rinsability, 7) non-corrosiveness, 8) stability during storage. Formulations of two solvent-type paint removers are cited. Wetting agents promote wetting power and penetration of the paint film; examples include petroleum sulphinate and ethylene glycol monobutyl ether. Cresylic acid is suggested as a paint stripper from aluminum, zinc and cadmium.—RPI.

#### 5.9.2, 5.9.3, 2.6

**Cleaning and Preparation of Metals for Electroplating. II. Soiling and Cleaning Procedures.** HENRY B. LINFORD AND EDWARD B. SAUBESTRE. *Plating*, 38, 367-375 (1951) Apr.

Nature of the problem of soiling and cleaning metals in the laboratory is reviewed. The cleaners, soils, and metals to be used are presented. The shape and size of panels used and the equipment necessary for the preparation of the samples are discussed. A standardized process for pre-cleaning, soiling, and cleaning panels prior to testing is described. Gravimetric data on amount of soil present under standard conditions are given, and effect of several operating variables is presented and analyzed.—BLR.

#### 5.9.2, 8.10.5

**Ultrasonics Has Wide Industrial Applications.** *Product Engineering*, 20, 153-154 (1949) Nov.

Outlines various present and potential metallurgical and nonmetallurgical applications. Applications of ultrasonics in caustic cleaning solution reduces time required for cleaning from 20 to 10 seconds.

#### 5.9.3

**Faster Cleaning of Metal Parts.** *Amer.*

*Exporter Industrial*, 147, No. 6, 24-26 (1950) Dec.

A description of compressed air cleaning equipment which uses an abrasive suspended in a liquid. It is marketed under the name of Hydro-Finish. It is claimed that the process may be used for deburring, for scale removal, for the preparation of surfaces to be metal-coated, for finishing threaded sections and for polishing die casting and other dies. The method is especially suitable for work on complicated shapes.—ZDA.

#### 5.9.3, 2.6

**Attack-Polish Method of Metallographic Preparation.** D. BOYD METZ AND HARRY W. WOODS. *Sylvania Electric Products, Inc.* Oct. 30, 1950. 11 p. (SEP-42).

A method has been developed for the preparation of all types of metallographic specimens. The procedure consists of a conventional mechanical polish with 2/0 or 3/0 jewelers' paper, followed by wheel polishing using a mixture of a common etchant and polishing powder. This etch-polish method was first used on wolfram and has now been successfully applied to uranium, beryllium, and zirconium. The method has less tendency to deform the surface than does the usual polish procedure or the electrolytic method. In addition, there is a considerable saving in the length of time necessary for the preparation of the samples plus the advantage of extreme simplicity of operation. The polished surfaces obtained by this method are free from disturbed material and, as viewed under high magnification, they give excellent definition of voids, inclusions, grains, and grain boundaries. In addition, it is possible to examine the specimens on the edges if so desired.—NSA.

#### 5.9.3, 3.5.8, 6.2.3

**The Effect of Shot Peening, and Shot Peening and Stress Relief, on the Fatigue Properties of SAE 1080 Steel.** N. B. BROWN. Paper before Can. Inst. Mining Met., Annual General Meeting, Ontario, April, 1950. *Trans. Can. Inst. Mining Met.*, 54, 121-125 (1951); *Can. Mining Met. Bull.*, 44, No. 467, 191-195 (1951) Mar.

The effect of a low-temperature stress relief at 232.2° C. on the fatigue properties of shot peened SAE 1080 steel was investigated. Specimens of the steel were first annealed. One-half of the steel bars were then normalized and the other half were water-quenched and drawn. Specimens of the steel with each heat treatment were tested in tensile notched bar impact and fatigue. The fatigue specimens were tested in air and water in R. R. Moore high-speed rotating bending machines in the un-peened, peened and peened-and-stress-relieved at 232.2° C. conditions. The endurance life and fatigue properties of specimens given a stress relief heat treatment after shot peening were superior to those of specimens given a smooth machine finish or shot peened. Tables of mechanical and fatigue properties, and corrosion in water, S-N curves and 5 references are given.—INCO.

#### 5.9.4, 5.4.5, 6.4.2

**MHC Hard-Coating Process for Aluminum.** *Light Metal Age*, 8, No. 7 & 8, 18, 29 (1950).

Some further details are given of the "MHC" new hard-surfacing process for aluminum and its alloys developed by

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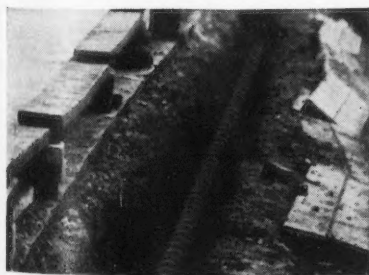
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### 5.9.4, 5.8.3

**New Developments Noted in Corrosion-Resistant Coatings.** *Steel*, 125, 90-94+ (1949) July 11.

News report on 95th convention of Electrochem. Soc. in Philadelphia, May 4-7, 1949 includes an extensive abstract of article on "Corrosion Resistant Coatings by Salt-Bath Chromizing" by L. E. Campbell, V. D. Barth, R. F. Hoechele and B. W. Gooser of Battelle Memorial Inst. It deals largely with this particular process and its limitations. There is a brief abstract of "A Unified Mechanism of Passivity" by R. R. Mears.

### 5.9.4, 5.9.2, 5.9.3

**Why Pre-Treatment?** P. HELLBERGER. *Product Finishing*, 4, No. 2, 40-45 (1951).

Pre-treatments of metals are classified under two headings, 1) metal cleaning and 2) surface film formation, e.g., phosphatizing. The importance of choice pre-treatment is emphasized and discussed with reference to heat-convertibility. It is suggested that convertible coatings require clean surfaces and that non-convertible films give better results on pre-treated surfaces.—RPI.

### 5.9.4, 6.3.14

**The "Protecta-Tin" Process: An Anti-Rust and Anti-Stain Treatment for Food Tins and Other Tinplate Containers.** *Product Finishing* (London), 3, No. 12, 58-61 (1950).

Full details are given for the operation and control of the "Protecta-Tin" process, which is claimed to increase the resistance of tinplate to rusting and to staining by sulfur products. The treatment does not affect the appearance of the metal and does not interfere with subsequent lacquering or lithographic printing. Processing is carried out by immersion for less than 30 seconds at 85° C. in a solution containing 9 lb. trisodium phosphate (anhydrous), 8 lb. sodium dichromate, 20 lb. sodium hydroxide, 3 lb. wetting agent ("Tee-pol") and water to make 100 Imperial gallons. The components are then thoroughly hot-water rinsed and over-dried.—MA.

<sup>1</sup> (120.94 U. S. gallons.)

### 5.9.4, 6.4.2

**The Theory of Phosphatizing (and the Successful Phosphatizing of Aluminum).** A. WUSTEFELD. *Arch. Metallkunde*, 3, No. 7, 253-255 (1949).

The characteristic features of phosphatizing in acid and in alkaline solutions are described, and, in both cases, are shown to be based on the same fundamental principles. Colloids form in the complex solutions by removal of the excess hydrogen or hydroxyl ions, such colloids having an opposite charge to that of the metal being attacked. They then discharge at the metal surface, and form a thin, well-bonded layer, which gradually builds up. It is difficult to phosphatize aluminum in acid baths as owing to the solution of the aluminum.



floucculation, and the rapid decrease in pH, aluminum phosphate colloids tend to lose their positive charge before they reach the metal surface. Successful treatment of aluminum can be carried out, however, if the following process is used: 1) degrease and pickle in a 5 percent sodium hydroxide solution at 50° C. for 5 minutes; 2) wash with cold water; 3) treat for 15 minutes in a solution containing hydrated aluminum sulfate 111.0 g./l., zinc phosphate 1.98 g./l.; 84 percent phosphoric acid-11.7 ml./l., 20 percent sodium hydroxide-306 ml./l., anhydrous sodium carbonate 7.3 g./l., powdered cupric oxide 0.25 g./l., and 2 percent mepasin-sulph-amino-acetic acid 5.0 ml./l.; pH 11.4; and 4) wash with cold water, and dry. The zinc and sodium salts in the phosphating solution act as accelerators, the cupric oxide as a depolarizer.—MA.

594, 6.4.2

**Metal Coloring.** C. HARRIS. *Metal Ind.* (London), 78, No. 10, 183-185 (1951) Mar. 9.

Methods of treatment for aluminum and its alloys. Polishing, chemical coloring, the brightening processes, anodizing, Bengough-Stuart, sulfuric acid and oxalic acid processes, dyeing anodized aluminum and immersion protection processes are discussed. In the brightening process using 70-75 phosphoric acid and 25-30 sulfuric acid, stainless steel tanks are employed. In dyeing anodized aluminum, the dye bath is made of stainless steel, glass or enamelled iron.—INCO.

594, 6.4.2

**Surface Finishing of Aluminum and Its Applications in the Aluminum Utensil Industry** (Oberflächenbehandlung von Aluminium und ihre Anwendung in der Leichtmetallwarenindustrie). J. ROLLE AND H. NEUNZIG. *Eberstadtler Offertenblatt* (Germany) 71, No. 22, 953 (1950) Nov. 25.

Aluminum utensils are usually made from commercial purity aluminum or copper-free aluminum-magnesium or aluminum-magnesium-silicon alloys. A copper content results in corrosion and discolorations. For most aluminum utensils, a finishing operation providing a protective coating is not necessary and most of them are etched. The requirements of an even but not too deep etch are met by a nitric-fluoric acid mixture. A matte finish is obtained after a brief treatment and the polish of the surface remains almost unchanged. Chemical and electrolytic oxidizing processes such as the MBV or EW processes have found little use for the finishing of aluminum utensils in Germany. Chemical oxidation is used only to provide protection for the inside of containers or as a base for lacquering. Anodic oxidation produces hard, corrosion resistant films of 0.01-0.02 mm. thickness. The higher the purity of the aluminum, the more clear and reflective the oxide coating will be. Gleaming surfaces may be obtained with "Raffinal" which is aluminum of the highest purity containing less than 0.01 percent iron and silicon and "Reflectal" with up to 2.5 percent magnesium. Reflectal (0.5-2.5 percent magnesium) is used more than Raffinal because it is stronger and is easier to fabricate than the higher purity material. Aluminum surfaces which are electrolytically or chemically polished must be anodized for protection, but this has little effect on the high polish. The Ertwerk chem-

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ical polish is simple and requires only 15-30 seconds while the VAW electrolytic polish requires approximately 10 minutes. Variations of the Eloxal process can produce chrome-, silver-, nickel-, or bronze-colored finishes on aluminum without dyeing. The oxide coatings are superior to nickel- or chrome-plated finishes in that the danger of galvanic corrosion resulting from a damaged surface is avoided. Polished Raffinal and Rectifal utensils do not affect the flavor of food, do not lose their polish, and do not discolor.—ALL.

## 6. MATERIALS OF CONSTRUCTION

### 6.2 Ferrous Metals and Alloys

#### 6.2.3, 3.2.2, 8.4.3

**How Hydrogen Attacks and Damages Steel in Refinery Equipment.** R. T. EFFINGER, M. L. RENQUIST, A. G. WACHTER and J. G. WILSON. *Oil Gas J.*, **50**, 99-100+ (1951) May 17.

Presents facts concerning the phenomenon by which hydrogen damages steel in conventional refinery equipment. Limited to the attack which occurs with the types of low-carbon steel commonly utilized in the construction of refinery-process equipment and piping. It is also limited to the attack associated with aqueous corrosion, in which the hydrogen which penetrates the steel is a product of the electrolytic corrosion cell. 32 references.—BLR.

#### 6.2.3, 4.4.1

**Adsorption of Polar Organic Compounds on Steel.** E. L. COOK and NORMAN HACKERMAN. *J. Phys. & Colloid Chem.*, **55**, 549-557 (1951) Apr.

Adsorption from solution of higher-molecular-weight aliphatic acids, amines, alcohols and certain esters on SAE-1020 steel powder with a specific surface area of 0.10 sq. m. per g. was studied. Two types of adsorption, irreversible and reversible, were observed for these systems. Extent of total adsorption was a function of molecular weight and polar group. It was found to exceed the calculated amount necessary for a complete close-packed monolayer by 20-70 percent depending on the compound. Points out importance of the results to corrosion prevention and lubrication. 11 references.—BLR.

#### 6.2.3, 5.3.4, 1.7.1

**Report of (ASTM) Committee A-5 on Corrosion of Iron and Steel (and Zinc-Coated) Products.** *Proc. Am. Soc. Testing Materials*, **49**, 110-127 (1949).

Annual report covering committee activities. Brief reports from sub-committees on methods of testing, sheet specifications, wire specifications, hardware specification, wire tests and hardware tests are included. In addition there is a detailed report covering 1948 inspections of field tests, wire and wire products after 12 years exposure at some 11 test sites. There was a wide difference in the loss of coating at different test locations, for example, the annual loss in ounces per square foot surface at Pittsburgh was .369, Sandy Hook .117, and State College .060.

#### 6.2.4, 3.7.4

**Contribution to the Study of Cast Irons With High Phosphorus Content.**

(In French.) JEAN GALEY. *Fonderie (Paris)*, **1951**, 2313-2326; disc. 2326-2328, Jan.

Presents a comprehensive study of constitution diagram, structure, mechanical properties, and corrosion resistance of cast iron, with high phosphorus contents. Results are tabulated and charted. They indicate that it is possible to obtain a fine homogeneous structure, mechanical properties being only mediocre; however, corrosion resistance to acids seems to be satisfactory. 18 references.—BLR.

#### 6.2.4, 4.6.11

**On the Corrosion Resistant Properties of Ferritic and Pearlitic Ductile Iron.** *Nickel Topics*, **3**, No. 9, 8 (1950) Sept.

Corrosion tests of limited duration on ductile (magnesium-containing) cast iron indicate that it can be applied in services where ordinary cast iron is satisfactory from a corrosion standpoint. It is no more difficult to cathodically protect ductile iron in sea water than gray iron. In neutral or alkaline environments such as sea water, ductile iron apparently has no marked advantage over plain cast iron, but is more resistant to acid attack when annealed to achieve a structure consisting essentially of ferrite and spheroidal graphite.

Corrosion rates in 5 percent sulfuric acid at 30° C. were 12.1 ipy for ductile iron compared with 6.5 ipy for gray iron; however, the corrosion rate of ductile iron heated 0.5 hour at 1750° F. and 5 hours at 1275° F. was 3.9 ipy compared with 8.2 ipy for similarly heat-treated gray iron. The ductile iron tested contained 2.90 percent total carbon, 2.55 percent silicon, 0.48 percent manganese, 0.08 percent phosphorus, 0.07 percent magnesium, and 1.44 percent nickel. Gray iron samples contained 3.25 percent total carbon, 2.92 percent silicon, 0.60 percent manganese, 0.08 percent phosphorus and 0.17 percent sulfur.—PDA.

#### 6.2.5, 2.3.2

**Intergranular Corrosion of 19-9 DL (TED NO. NBS 2562).** SAMUEL J. ROSENBERG. U. S. National Bureau of Standards, Report 12 to the Bureau of Aeronautics, June 1949.

Four commercial heats of austenitic stainless steel 19-9 DL were tested to determine susceptibility to intergranular embrittlement after annealing followed by sensitizing treatments. None of the steels were immune to intergranular attack although the severity varied with type of annealing. An anneal at 1650° F. followed by air-cooling was the least damaging. The boiling solution used to develop intergranular embrittlement frequently caused considerable corrosion.

Sheet steel, hot-rolled from 2000° F. and finished at 1200-1400° F. to 0.050-0.055-in. thickness was sensitized by heating for 8 days at 1000° F. or 2 hours at 1200° F. The condition of the steel before sensitizing was 1) as received, 2) annealed 30 minutes at 1650° F., 3) annealed 30 minutes at 1800° F., or 4) annealed 3 minutes at 1975° F. Annealing was followed by air cooling. The sensitized samples were then boiled in a solution consisting of 10 g copper sulfate pentahydrate, 10 ml sulfuric acid, and 90 ml distilled water for 14 days unless failure occurred earlier. Intergranular corrosion was evaluated by increase in electrical resistivity, loss of metallic ring, cracking after bending,

and metallographic examination. The un-etched microstructure of the four steels showed angular insoluble particles in varying amounts; the steel containing the largest amount showed the least intergranular corrosion.

Tables give analyses of the steels and the various corrosion results.

#### 6.2.5, 3.5.9

**Russians Have New Heat-Resistant Alloys.** KORNILOV (Translated by S. L. CASE). *Reports Acad. Sci. USSR. Iron Age*, **167**, No. 12, 65-69 (1951) Mar. 22.

Shortages of nickel and cobalt in Russia during World War II stimulated development of iron-chromium-aluminum alloys as a substitute for nickel-chromium in electrical resistors and other high temperature uses. Results of tensile and hardness tests on ternary alloys containing up to 10 percent aluminum and 30 percent chromium show that regardless of the chromium content, aluminum raises the hardness of the alloys, and that chromium is also a strong hardening element. The electrical resistance of iron-chromium alloys increases with increasing chromium and aluminum contents, but aluminum exerts a stronger effect. They show a high resistance to scaling. Scaling losses are under 1 gram per square meter per hour, which is considerably less than those encountered in heat-resisting chromium-nickel alloys. The resistance of iron-chromium-aluminum alloys to scaling is sharply deteriorated by an increase in their carbon content. The author found three regions of commercially useful alloys. Alloys containing 15-30 percent chromium and 2-10 percent aluminum are heat resistant up to 2280° F. and can be hot rolled and cold drawn. Alloys containing 35-45 percent chromium and 5-12 percent aluminum are heat resistant to 2450° F. and can be hot worked only. Alloys containing 65-75 percent chromium and 7.5-12.5 percent aluminum are heat resistant up to 2730° F. and are very brittle and cannot be deformed either hot or cold. Property tables, a table showing the heat resistance of iron-chromium-aluminum alloys as compared with Nichrome and graphs on electrical resistivity, notched bar toughness and scaling are given.—INCO.

### 6.3 Non-Ferrous Metals and Alloys—Heavy

#### 6.3.1, 1.7.1

**Report of (ASTM) Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.** *Proc. Am. Soc. Testing Materials*, **49**, 150-151 (1949).

Annual report outlining briefly activities of various sub-committees. The sub-committees mentioned are Spray Testing, Weather, Galvanic and Electrolytic Corrosion. Contribution from Weather Committee is appended to report. This article is entitled: "Effect of Weather on The Initial Corrosion Rate of Sheet Zinc" by O. B. Ellis.

#### 6.3.4, 4.3.3

**Research Toward Development of Noncorrosive Metals and Magnetic Alloys, and the Production of Magnetic Alloys by Powder Metallurgy.** WALTER BECK, Lehigh University. Final Report: U. S. Signal Corps Contract W6-039-sc-32033, November 1950. viii, 139.1, tables, diagrs.

October, 1952

This report covers progress during the last quarter year of this contract and also summarizes the results obtained during the course of the entire project. Topics included in the corrosion phase are: dissolution of aluminum and aluminum alloys in alkali chloride solutions, effect of heat treatment of aluminum alloys, dissolution of magnesium in hydrochloric acid, corrosion resistance of magnetically soft materials, behavior of commercially pure aluminum in neutral potassium chloride solutions under accelerated conditions, and influence of oxygen concentration on corrosion of commercially pure aluminum in neutral potassium chloride solutions. Tables and diagrams are included.

PDC Comment: All progress reports of this project are on file at the Center. Important results and conclusions on the corrosion phase of the project have been abstracted [Quarterly Reports 6, 8, and 11 (December 1947, July 1948, December 1948); PDA 7; Met 53, G-2832(11B).]—PDA.

## 63.4, 63.3, 3.5.9

**Quarterly Progress Report on Alloys for High-Temperature Service, to Office of Naval Research.** E. E. FLETCHER, A. R. ELSEA, A. B. WESTERMAN, AND G. K. MANNIN. Battelle Memorial Inst., June 16, 1950. 21 p. (NP-1725).

The investigation of the effects of tungsten and molybdenum on the  $\alpha$ - $\beta$  transformation temperature range in cobalt-chromium alloys was continued. Work was continued on the production of satisfactory carbon bearing ternary alloys for use in determining the effect of carbon on the  $\alpha$ - $\beta$  transformation temperature range. Early tests resulted in excessive carbon losses during homogenization. The heat-treating technique and apparatus were modified; as a result, several carbon bearing alloys have now been homogenized with only a slight loss of carbon. Commercially produced alloys, Stellite 21 type, of four different analyses have been homogenized and the carbon was retained quite satisfactorily. Partial melting occurred when the usual homogenizing treatment of 80 hours at 1260° C. was used. Some modification of this treatment may be required for the Vitallium-type alloys and for the carbon bearing ternary alloys, especially those with relatively high carbon contents. Fourteen creep-rupture tests at 780, 915, and 1000° C. have been completed on cobalt-chromium alloys containing nitrogen.—NSA.

## 63.8

**Dross Formation of Lead Melts in Calm Air.** (In German.) W. HOFMANN AND K. H. MAHLICH. *Werkstoffe u. Korrosion*, 2, 55-68 (1951) Feb.

Describes experiments on effects of temperature, furnace atmosphere, and alloying additions on formation of dross from soft lead and its alloys. A new method of retarding this effect consists of heat-treating the melt at 500-700° C. with frequent stirring. Graphs and tables are presented. 25 references.—B.J.R.

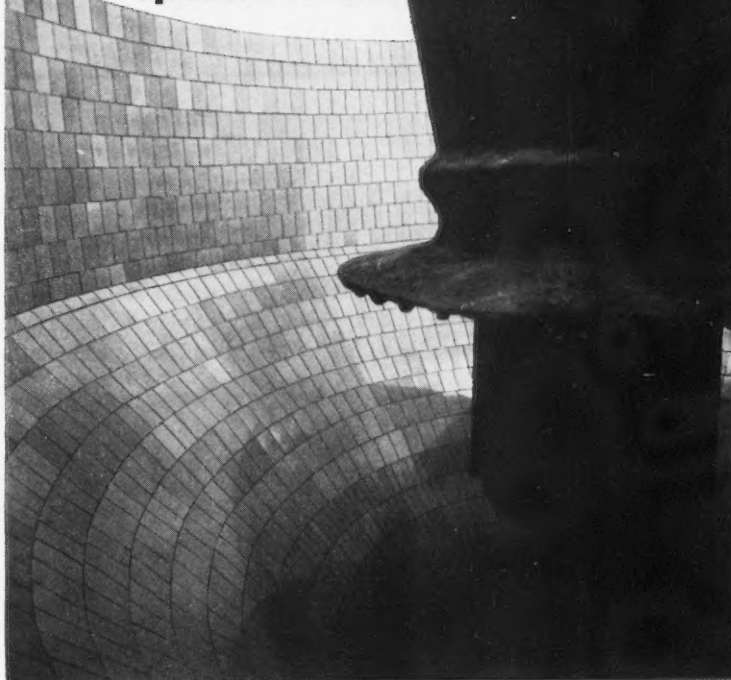
## 63.9, 5.9.4

**Protective Coating for Molybdenum.** *Products Finishing*, 15, No. 3, 60-62 (1950) Dec.

Work conducted at Battelle Memorial Institute on vapor deposition of refractory-type metals on both metal and non-metal surfaces is briefly described.

Coatings of molybdenum and tungsten are formed by reduction of their chlorides.

## INSTEAD of expensive alloys and special steels



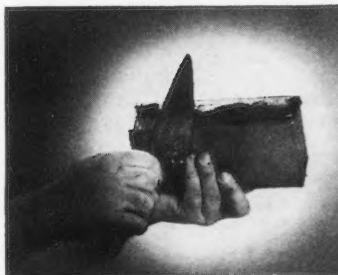
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## Technical Service Data Sheet

**Subject: HOW TO MAKE PAINT STICK TO GALVANIZED IRON WITH LITHOFORM®**

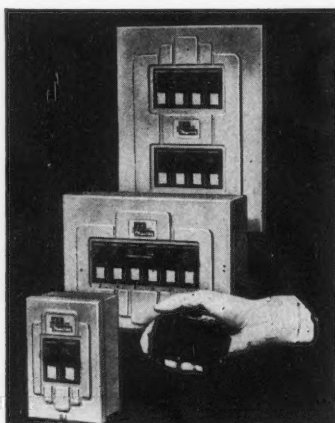
### INTRODUCTION

"Lithoform" forms a dense, zinc phosphate coating on zinc, cadmium, and galvanized surfaces—including Galvanneal, cadmium plated steel, zinc plated steel, zinc base alloys, and zinc base die castings. The "Lithoform" coating, which is non-metallic and inactive, retards reaction between alkaline metal oxide and the paint film. Peeling and loss of adhesion are thus greatly retarded on painted Lithorized zinc and cadmium.

### ADVANTAGES OF "LITHOFORM"

"Lithoform" forms a durable bond for paint. It is economical. It eliminates frequent repainting. It protects both the paint finish and the metal underneath. "Lithoform" meets these Government Finish Specifications:

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MIL-E-917A (Ships)  
JAN-F-495  
AN-F-20  
U.S.N. Appendix 6



Photograph by courtesy of Murray Manufacturing Corp.  
Murray Circuit Protectors are fully magnetic and provide maximum protection for both domestic and industrial wiring. Housings are of galvanized iron which is Spray Lithorized for long paint life.

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"Lithoform" can be applied by brushing or spraying the work with simple hand equipment, by dipping it in tanks, or by spraying it in industrial power washers.

**Brush.** Galvanized bay windows, cornices, rain gutters, hardware, building siding, truck panels, and farm equipment are typical of the many surfaces that are treated effectively with Brush "Lithoform".

**Dip.** This grade is used for coating cleaned surfaces of such typical products as cabinets, refrigeration condensers, etc., immersed in heated solutions in tanks.

**Spray.** The spray process is the most logical one with which to coat sheets, coiled strip or duplicate products best processed on a conveyor.



WRITE FOR FURTHER INFORMATION ON "LITHOFORM" AND ON YOUR OWN METAL PROTECTION PROBLEMS.



rides. The platinum group metals are deposited by decomposition of their carbonyl chlorides. Tantalum and columbium are readily deposited on both metal and nonmetal bases by hydrogen reduction of their pentachlorides at temperatures above 600° C. On refractory bases such as molybdenum, tungsten, and graphite, thermal decomposition of the pentachlorides and pentabromides produces coatings of these metals. Vanadium coatings are obtained by decomposing vanadium diiodide at 1000-1200° C. on the surface of the base metal.

Coatings of refractory nitrides, borides and silicides are obtained by reacting the metal chlorides with nitrogen-hydrogen, boron trichloride-hydrogen, and silicon tetrachloride-hydrogen atmospheres, respectively, at a heated surface under controlled conditions.

A highly corrosion-resistant coating of molybdenum disilicide on molybdenum metal was obtained by treating the metal at 1000-1800° C. with a silicon tetrachloride-hydrogen atmosphere. Coatings 1 mil thick completely protected the base metal for more than 4000 hours in air at 1000° C. and for more than 30 hours at 1700° C. Thicker coatings gave longer protection. Although brittle at room temperature, the coatings have limited ductility at red heat; they also tend to be self-healing.

By proper selection of reaction and plating conditions, alloys of most of the refractory metals can be obtained over wide ranges of composition, enabling alloys not obtainable by conventional methods to be prepared.—PDA.

#### 6.3.10, 3.8.3

**Phase Boundary Potentials of Nickel in Foreign Ion Solutions.** D. MACGILLAVRY, J. J. SINGER AND J. H. ROSENBAUM. Clark Univ. Paper before ACS, Gen. Mtg., Div. of Phys. & Inorg. Chem., Chicago, Sept. 1950. *J. Am. Chem. Soc.*, 73, No. 3, 5388 (1951) Mar.

Stable potentials could be obtained within 5-15 hours in a series of potassium hydroxide solutions, and in a series of phosphate buffer solutions, covering the entire pH range. The stationary potentials, calculated against the standard hydrogen electrode, are plotted as a function of the pH of the solutions. Since aeration greatly accelerates the corrosion of nickel in acid media, oxygen was excluded from the half cells. Lengths of nickel wire of high purity were thoroughly cleaned and pretreated. The samples were freed from oxides and gases by induction heating first in hydrogen and then in a vacuum. The solutions were freed from oxygen before making contact with the nickel samples. After each run the solutions were tested for nickel which might have gone into solution. The results were always negative, even in the acid phosphate solutions, unless oxygen had been admitted.—INCO.

#### 6.3.11, 4.3.1, 4.4.1

**Hydrochloric Acid versus Construction Materials.** E. F. ROSENBLATT. Symposium. *Chem. Eng.*, 56, 243-244 (1949) Feb.

A portion of Part III of Symposium deals briefly with the activity of precious metals—silver, gold, palladium, and platinum. All of these metals show some activity in the presence of oxidizing agents. All are satisfactory in the absence of oxidizing agents. Platinum is more resistant than the others.

# *Positive* CORROSION PROTECTION

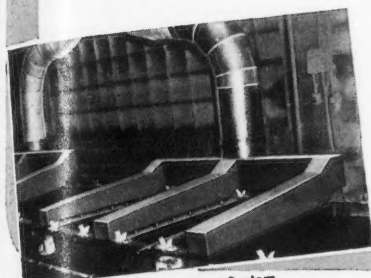
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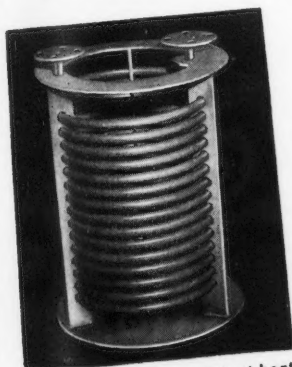
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# Understanding Corrosion:

## THE EFFECT OF HUMIDITY

**R**elative humidity — the degree to which air is saturated with moisture — influences corrosion in one direction where metal is exposed to atmosphere and in another where it is immersed in a solution surface-exposed to the same atmosphere.

It is humidity, in combination with temperature, that determines whether moisture can exist on a metal surface. Because moisture is essential to atmospheric corrosion, humidity, at normal temperatures, becomes the factor that determines whether corrosion will occur and to what extent. Its effect, negligible below 30%, increases until a critical point is reached at about 65%. When air so moisture-laden carries even minute quantities of such gases as sulphur dioxide, corrosive action is greatly intensified. It is this condition that renders difficult the protection of steel stacks, breechings and economizers in service below the dew point.

For metal immersed in a corrosive solution, the reverse is true. More rather than less humidity in

the air above is the preferable situation because of the way in which humidity influences the rate of oxygen solution. The rapid evaporation that occurs in dry atmosphere leaves a cool, dense surface layer of liquid that carries dissolved oxygen through the solution as fresh, unsaturated liquid replaces it for a repetition of the process. As the pace at which oxygen is brought in contact with the metal is thus stepped up, corrosion increases proportionately.

Protection of metal exposed to atmosphere or immersed in corrosive solutions, particularly under difficult or unusual service conditions, has long been the function of Dampney equipment-engineered coatings. Our experience in the handling of corrosion problems peculiar to industrial power and processing operations is not only extensive but specific . . . applicable, we feel sure, to your individual needs. Let us tell you more about Dampney coatings and what they offer . . . in terms of your requirements.



155-1

HYDE PARK, BOSTON 36, MASSACHUSETTS

6.3.14, 3.5.9, 2.3.7, 6.3.1

**A Study in Non-Ferrous and Ferrous Alloys at Low Temperatures.** Research Investigations Leading to the Development of Improved Non-Ferrous and Ferrous Alloys. MILTON MARGOLIS AND ALFRED BORNEMANN, Stevens Institute of Technology. Final Report; U. S. Signal Corps Contract W-36-039-sc-38135, November, 1950.

Tensile, impact, and fatigue tests at temperatures down to  $-100^{\circ}\text{F}$ . were made on pure tin, tin-lead alloys, commercial solders, Easy-Flo silver solder, two tempers of phosphor bronze, and soldered joints. Several tin alloys were stored as long as one year at  $-40^{\circ}\text{F}$ . and  $-100^{\circ}\text{F}$ . to study their susceptibility to 'tin disease', i.e., the tendency of tin atoms to pass from the body-centered tetragonal lattice to the diamond-type lattice as the temperature is lowered below the transition point ( $55.8^{\circ}\text{F}$ ).

Silver solder and phosphor bronze had good engineering properties at temperatures as low as  $-100^{\circ}\text{F}$ ; Easy-Flo silver solder appeared satisfactory as low as  $-70^{\circ}\text{F}$ . Contrary to previously published opinions, neither lead, bismuth, nor antimony completely retarded the transformation of tin to the low-temperature nonmetallic form. One year of low-temperature storage, however, caused no serious impairment of the physical properties of tin-base solders. Strain such as that induced in fatigue testing greatly accelerated the transformation of pure tin.

The tin-base alloys stored at low temperature included 50 percent tin—50 percent lead, with and without 0.5 percent antimony and/or 0.1 percent bismuth. The commercial tin solders examined contained 0.38 percent bismuth, 0.69 percent antimony, and 40 percent lead.

Equipment for the various low-temperature tests is described.

6.3.15, 2.3.5, 3.6.6

**Investigation of the Corrosion Resistance of Titanium and Titanium Alloys.** J. H. JAMES, U. S. Naval Air Material Center, Philadelphia. Report AML NAM AE 411033, Part I February 1950.

The solution potential of titanium indicates that it is cathodic to most metals used in aircraft construction and therefore suffers less attack than the metals with which it comes in contact in corrosive electrolytes. Surface treatment of titanium reduces its solution potential. The potential measurements also indicate that nascent hydrogen and oxygen do not enter the metal lattice at room temperature.

The weight losses of dissimilar metals coupled to titanium in sodium chloride solution are comparable with those suffered by these metals in contact with 18-8 stainless steel. The corrosion rates of titanium and stainless steel in sodium chloride are as low as 0.23 and 0.50 mg per sq. dm. per day, respectively, and are not changed significantly when connected to dissimilar metals.

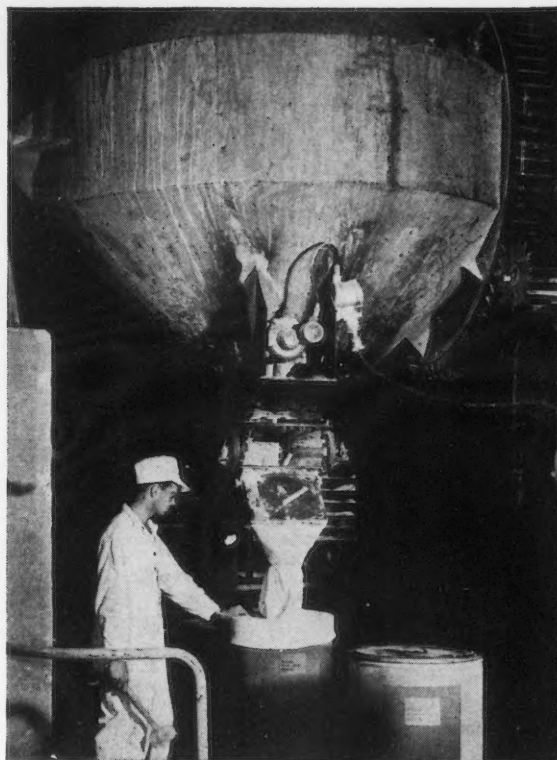
Contacts of titanium or 18-8 stainless steel do not accelerate the corrosion of zinc, zinc anozinc treated, cadmium, and cadmium-tin plated steel in salt spray, high humidity, or atmospheric exposures, as indicated visually.

Titanium can be anodized in chromic and sulfuric acid electrolytes in a manner similar to aluminum. The outer surface of the oxide coating formed is nonadherent. The adhesion of paint to titanium is improved by anodizing if zinc chromate



# How Nickel Protects Saran Purity

... from wet polymer  
to  
finished plastic



Saran molding powder is mixed and made ready for shipment in nickel blenders designed and manufactured by Industrial Process Engineers, Newark, N. J. The 220 cu. ft. blenders have conical sections of solid nickel and nickel-lined cylindrical sections. Nickel was used because other metals have proved detrimental to the quality of the plastic.

As a finished product, saran is a wonderful material—it's inert to most oils and chemicals at room temperatures—has high tensile and impact strength.

But it must be handled gently in certain stages of production.

The manufacturers of the saran powder found that out in the processing . . .

As a wet polymer it is sensitive to many metals and may turn yellow when it comes in contact with them. The chemical company which makes the powder found a solution to this problem in specially designed blenders made of nickel.

Then the manufacturers of saran-molded products discovered that certain metals catalyze the decomposition of the material. They too

found that they could guard against this and protect the purity of the hot plastic by using nickel and Inco Nickel Alloys for all parts the plastic came in contact with.

And when the extruders found that the hot saran was corroding their equipment, they turned to tough Duranickel for cylinder liners, screws, dies and other parts.

Perhaps you, too, have a problem of purity-protection or corrosion. If so, why don't you write Inco's Corrosion Engineering Section and tell them your problem. They'll be glad to help you.

Right now, of course, Inco Nickel Alloys are on extended delivery because of defense needs. Therefore, orders should be placed well in advance, giving necessary NPA rating and complete end-use information.

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"S"® MONEL • NICKEL • LOW CARBON NICKEL • DURANICKEL®  
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Nicolet Industries, Inc.	24
Pennsylvania Salt Mfg. Co.	42
Pipe Line Service Corp.	ix
Pittsburgh Coke & Chemical Co.	xi
Plastic Engineering & Sales Corp.	20
Polyken Industrial Tapes, Division of Bauer & Black	7
Positions Available and Wanted	30
Reilly Tar & Chemical Co.	3
Richardson-Allen Corp.	26
Saran Lined Pipe Co.	48
Service Engineers, Inc.	11
Stearns, D. E., Co.	Back Cover
Tapecoat Co., The	49
Tretolite Company	25
United States Stoneware Co.	23
Wilbur & Williams Co.	ix
John Wiley & Sons, Inc.	22
Williamson, T. D., Inc.	29
Wright Chemical Corp.	5

primer AN-P-656 is applied. Excellent paint adhesion is obtained on unanodized titanium when the priming coat is WP-1 metal conditioner; this adhesion is not improved by anodizing.

The metals employed for the dissimilar couple tests included 75S-T6, ALC 75S-T6, 24S-T3, 2S-, 3S-H14, 52S-H14, and 61S-T aluminum alloys, 60-40 brass, cadmium, chromium, copper, 4130 steel, Inconel, J1H magnesium, nickel, lead, tin and zinc. Solution potentials for these metals were determined after 1, 30, and 60 minutes of immersion, using a Brown recording potentiometer and a 0.1 N calomel reference electrode.

The titanium used was unalloyed. Its solution potentials were determined over a 24-hour period in the untreated state and after immersion treatments in 1 N hydrochloric, nitric, sulfuric, or acetic acid, or after hydrogen treatment which involved electrolyzing the titanium samples as cathodes for 15 minutes in 5 percent potassium carbonate solution at 85° F. and 1 amp per sq. dm. current density. Some specimens were anodized for 15 minutes in 5 percent chromic acid at 95° F. and 60-v potential.—PDA.

## 6.3.15, 5.9.4

**Corrosion Resistance of Anodized and Unanodized Titanium—Effect of Common Mineral Acid Solutions.** C. MA, E. M. PERES, Tulane Univ. *Ind. and Eng. Chem.*, 43, 675-679 (1951) Mar.

Corrosion rates of anodized and unanodized titanium in acid solutions of various strength were determined by the alternate immersion corrosion test at 35° C. In addition, the behavior of anodized and unanodized titanium in selected acid solutions at the boiling points were investigated by the total immersion corrosion test, without aeration. It was necessary to evaluate the protective films formed under various conditions since no titanium anodization condition was available. Corrosion resistance of titanium depends primarily on the oxidizing or reducing tendencies of the corrosive medium in which it is immersed. In oxidizing media, both anodized and unanodized titanium were equally resistant to attack at all concentrations and temperatures tested. In reducing media, both were readily attacked at about the same rate and in neutral media the anodized metal was seen to be superior in resisting attacks by acids. The passivation of titanium depends upon a tight oxide film developed on the metal surface. Tables and graphs are included. 12 references.—INCO.

## 6.7 Duplex Materials

## 6.7.2, 5.4.2

**High-Temperature Protection of a Titanium-Carbide Ceramal With a Ceramic-Metal Coating Having a High Chromium Content.** DWIGHT G. MOORE, STANLEY G. BENNER, AND WILLIAM N. HARRISON. National Advisory Committee for Aeronautics, Technical Note 2329, Mar. 1951, 31 pages. (TL570 Un3t.)

Describes the development of the above. The ceramal contained 80 percent titanium carbide and 20 percent cobalt. The coating, designated A-479M, contained 80 percent chromium and 20 percent alkali-free frit (glass). It was prepared as a slip, with clay as floating

agent, and applied to the ceramal by dipping or spraying then firing at 2000° F. for 10 minutes in a purified H<sub>2</sub> atmosphere. Laboratory tests and microstructure studies indicated that this coating has adequate adherence and thermal shock resistance and that it would be expected to creep at a temperature as low as 1500° F. At a blade temperature of 1800° F., this coating would probably greatly prolong the life of a K-138 turbine blade under operating conditions sufficiently severe to accelerate failure of the uncoated blade by oxidation. Photographs, tables, and graphs are included.

## 7. EQUIPMENT

## 7.1 Engines, Bearings and Turbines

## 7.1, 8.4.3

**Lubricated Plug Valves for Drilling and Wellhead Services.** E. E. HEDENE, Rockwell Mfg. Co. Paper, ASME, Petrol. Div., New Orleans, Sept. 27, 1950. *Petroleum Engr.*, 22, No. 13, B59, B61-64 (1950) Dec.

Basic requirements to be met with oil field valves and the major design features of lubricated plug valves developed to meet these requirements. Rigidity, lubrication, seating thrust, corrosive oil field service, welded hard facings and oil field valve designs are discussed. 12-14 chromium steel entirely resists the type of well-head corrosion found in the Gulf Coast area but are expensive. 5 and 9 nickel alloy steels are being used in corrosive wells. Five nickel steel valves can be manufactured cheaper than 12-14 chromium steel, but 9 nickel steel valves are approximately the same price. The 9 nickel alloy is difficult to handle in the foundry. If there is any doubt, 12-14 chromium valves are recommended. Illustrations are given.—INCO.

## 7.1, 8.9.5

**Bearings and Lubrication for Marine Turbines and Reduction Gears.** F. C. LINN (G. E.) AND J. T. BURWELL (M. I. T.). Paper, Soc. Naval Arch. and Marine Eng., N. Y., Nov. 1950. *Marine Eng. & Shipping Rev.*, 55, No. 12, 47-54 (1950) Dec.

Developments in progress will increase pressures and temperatures, gear tooth loads, and loading on the bearings of the turbine and gears, and will allow for lower viscosity oils to be used in propulsion equipment. Journal and thrust bearings, bearing materials, the lubricant, gravity and pressure oiling systems and calculated bearing performance are discussed. Tin and lead base babbitt, aluminum (1 nickel), and trimetal bearings are considered. Tests on aluminum liners indicated that it is satisfactory but the shaft and lining scores much worse than the babbitts. Conventional refined oil allows rusting of uncoated ferrous metals if water is present. Inhibited oils are used for protection against rusting, oxidation and foaming. Diagrams of lubricating systems and bearings are given.—INCO.



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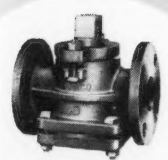
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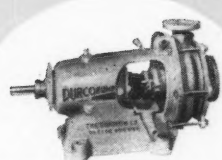
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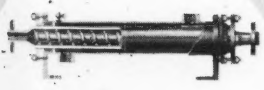
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